NATIONAL URANIUM TAILINGS PROGRAM

URANIUM TAILINGS REFERENCE MATERIALS

NUTP 21

Canada

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URANIUM TAILINGS REFERENCE MATERIALS

C.W. Smith *H.F. Steger* *W.S. Bowman*

National Uranium Tailings Program

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MICROMEDIA

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MICROMEDIA

FOREWORD

There are currently about 130 million tonnes of mine wastes (tailings) produced from the mining and milling of uranium ore that are on, or near, the surface in Canada. At public hearings and other forums held in connection with proposals for new uranium production facilities, concern has been expressed about the impact that these wastes may have on the environment in the future. Government regulatory agencies believe that there may be long-term environmental and health consequences associated with uranium tailings that are not yet fully understood.

As a result of a year-long study, undertaken by a group of experts from government, industry, and the universities, into the possible long-term problems of uranium tailings management, the decision was taken by the Federal Government to launch an R & D program to assist in clarifying the nature and extent of problems that may arise in the future. It is anticipated that the results of such a program will be of use to the Atomic Energy Control Board and to provincial regulatory agencies in the establishment of criteria for the decommissioning and abandonment of uranium tailings. To this end, the National Uranium Tailings Program of CANMET has been funded to the extent of $9.5 million for a period of five years.

The main objective of this Program is to provide a scientifically credible information base that will assist government regulatory agencies in making confident, knowledgeable decisions, and in establishing criteria for the long-term protection of the environment and human health. The scientific information base will, among other things, describe the chemical and physical processes that involve tailings constituents and their movement along pathways from the tailings into the biosphere.

The National Uranium Tailings Program's activities, which have been entirely contracted out, have included research into the processes involved in the behaviour and movement of contaminants from tailings, field sampling and measurements, chemical analyses, and predictive modelling. As an important part of the Program output, manuals have been prepared that incorporate the results of this work.

This manual has been prepared for the purpose of making available up-to-date information and data that will be of interest and practical use to those directly concerned with operational aspects of uranium tailings management and regulation.

W.G. Jeffery
Director General
Canada Centre for Mineral and Energy Technology

PREFACE

Quality assurance in the production of data is indispensable to the success of the National Uranium Tailings Program. In order to provide as much assistance as possible in ensuring high quality chemical analyses for radionuclides and other constituents in a uranium tailings matrix, standard reference materials were prepared to cover the range of tailings composition encountered in Canada. These reference materials will be used to maintain a high level of quality control for all work performed by contract on behalf of the Program, and will also be available to all others that may find them useful in verifying the performance of their own equipment and procedures.

As additional reference materials, relevant to the Program are certified in the future, supplements to this manual will be prepared and published.

ACKNOWLEDGEMENTS

At the beginning of the Program, Lloyd Dalton and Clint Smith of CANMET Chemical Laboratories served as Scientific Authorities on behalf of NUTP in the exchange of analyses with participating laboratories. This responsibility has subsequently been assumed by Mr. Roy John, Project Manager, Measurements, of the National Uranium Tailings Program Office. The laboratories that participated in the exchange program are listed in the body of the manual. The efforts of the following CANMET staff in organizing and operating the project in developing the reference standards are gratefully acknowledged: C.W. Smith, H.F. Steger and W.S. Bowman.

V.A. Haw
Director
National Uranium Tailings Program

URANIUM TAILINGS REFERENCE MATERIALS

by

C.W. Smith, H.F. Steger* and W.S. Bowman**

SYNOPSIS

Samples of uranium tailings from Bancroft and Elliot Lake, Ontario, and from Beaverlodge and Rabbit Lake, Saskatchewan, have been prepared as compositional reference materials at the request of the National Uranium Tailings Research Program. The four samples, UTS-1 to UTS-4, were ground to minus 104 μm, each mixed in one lot and bottled in 200-g units for UTS-1 to UTS-3 and in 100-g units for UTS-4. The materials were tested for homogeneity with respect to uranium by neutron activation analysis and to iron by an acid-decomposition atomic absorption procedure.

In a *free choice* analytical program, 18 laboratories contributed results for one or more of total iron, titanium, aluminum, calcium, barium, uranium, thorium, total sulphur, and sulphate for all four samples, and for nickel and arsenic in UTS-4 only. Based on a statistical analysis of the data, recommended values were assigned to all elements/constituents, except for sulphate in UTS 3 and nickel in UTS-4.

The radioactivity of thorium-230, radium-226, lead-210, and polonium-210 in UTS-1 to UTS-4 and of thorium-232, radium-228, and thorium-228 in UTS-1 and UTS-2 was determined in a radioanalytical program composed of eight laboratories. Recommended values for the radioactivities and associated parameters were calculated by a statistical treatment of the results.

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RÉSIDUS D'URANIUM, MATÉRIAUX DE RÉFÉRENCE

par

C.W. Smith, H.F. Steger* et W.S. Bowman**

SYNOPSIS

Des échantillons de résidus d'uranium provenant de Bancroft et d'Elliot Lake en Ontario et de Beaverlodge et de Rabbit Lake en Saskatchewan ont été préparés comme matériaux de référence de composition connue à la demande du Programme national de recherche sur les résidus d'uranium. Chacun des quatre échantillons, UTS-1 à UTS-4, a été broyé à une granulométrie inférieure à 104 μm et mélangé pour former un lot. Les résidus UTS-1 à UTS-3 ont été mis dans des bouteilles de 200 g et l'UTS-4 a été mis dans des bouteilles de 100 g. On a vérifié l'homogénité des matériaux quant à l'uranium par la méthode d'activation neutronique et quant au fer total par une méthode de décomposition acide et de spectrométrie d'absorption atomique.

En vertu d'un programme analytique de *libre choix*, 18 laboratoires ont soumis des résultats pour un, ou plusieurs des éléments/constituants suivants: fer total, titane, aluminium, calcium, baryum, uranium, thorium, soufre total et sulfate pour les quatre matériaux, et nickel et arsenic pour l'UTS-4 seulement. Après une analyse statistique des données, on a assigné des valeurs recommandées à tous les éléments/constituants, sauf au sulfate pour l'UTS-3 et au nickel pour l'UTS-4.

On a déterminé la radioactivité des isotopes, thorium-230, radium-226, plomb-210, et polonium-210 pour les quatre matériaux, et thorium-232, radium-228, et thorium-228 pour l'UTS-1 et l'UTS-2, dans le cadre d'un programme radioanalytique auquel huit laboratoires ont participé. Les valeurs recommandées pour les activités des isotopes et des paramètres associés ont été calculées à l'aide d'une analyse statistique des données.

**Chercheurs scientifiques, Laboratoires des sciences minérales, CANMET, Énergie, Mines et Ressources Canada, Ottawa, K1A 0G1.*

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INTRODUCTION

The preparation of compositional reference materials from several uranium tailings samples, typical of existing Canadian tailings sites, was initiated under the auspices of the National Uranium Tailings Research Program (NUTRP) in late 1982 (1). These were intended for use in quality-control functions for both chemical and radiochemical analytical measurements with the National Measurement Program of NUTRP. The reference materials were also to be made available through the Canadian Certified Reference Materials Project (CCRMP) for more general use by the analytical community.

CCRMP currently offers many compositional reference ores, concentrates, and related products, typical of Canadian deposits and not, in general, available from other sources for use in analytical laboratories associated with mining, metallurgy, and the earth sciences (2). Among these are uranium ores from Elliot Lake, Ontario, and Beaverlodge, Saskatchewan, of which four, DL-1a, DH-1a, BL-4a and BL-5, have been certified for radium-226 activity (3).

This report describes the preparation and characterization of the four uranium tailings samples: UTS-1, UTS-2, UTS-3 and UTS-4. The description of their nature, physical preparation, and estimation of the consensus values and related statistical parameters for use as reference materials follows.

The results of the analytical program to establish the concentration of total iron, titanium, aluminum, calcium, barium, uranium, thorium, total sulphur, sulphate, nickel and arsenic in the tailings samples are presented in "Part A" of this report.

The results of the radioanalytical program to determine the activity of thorium-230, radium-226, lead-210, and polonium for all four samples, and also for UTS-1 and UTS-2, thorium- 232, radium-228 and thorium-228, are presented in "Part B".

NATURE AND PREPARATION

UTS-1

The uranium tailings sample for UTS-1 was donated to NUTRP and CCRMP in October 1982 by Madawaska Mines Limited located near Bancroft, Ontario. The mine is in a belt of meta-gabbro and amphibolite. The ore is part of a zone of irregular pegmatitic granite dykes, with metasomatic phases. Uranium is present as uraninite and uranothorite with minor uranophane (4). The ore is comminuted to 50% minus 74 μm and is leached at pH = 1.6 to 1.8 with sulphuric acid and sodium chlorate under agitation with compressed air with a 24 h retention period. The mill tailings slurry is treated with lime to pH = 9 before disposal (5).

UTS-2

The tailings sample for UTS-2 was donated by Rio Algom of Elliot Lake, Ontario. The ore is a pebble conglomerate. The pebbles are mainly quartz with some chert. The matrix is a sericitic, feldsphathic quartzite containing about 10% pyrite on a whole ore basis (6). The comminuted ore is leached at 60-70°C at a concentration of 40 g/L sulphuric acid under agitation with compressed air for approximately 40 h. The mill tailings slurry is treated with limestone and lime to raise the pH to 10 before disposal (7).

UTS-3

The tailings sample for UTS-3 was donated by Eldorado Nuclear Limited at Beaverlodge, Saskatchewan. The orebody consists of complexes of disseminations and stringers, lenses and veins of pitchblende in reddish-brown mylonitized oligoclase saturated with dusty hematite. Pitchblende is the sole radioactive mineral present. Minor amounts of carbonates and pyrites occur along with the pitchblende (8). The pyrites are separated as a concentrate by flotation at pH 10 (sodium carbonate) using isopropyl xanthate. This concentrate is leached with 100 lb/ton (44 kg/1000 kg) of sulphuric acid and 2.5 lb/ton (1.1 kg/1000 kg) of sodium chlorate. The uranium is precipitated with magnesium hydroxide and sent to the carbonate leaching circuit.

The slurry of flotation tailings and the above precipitated uranium in 100 g/L dissolved carbonate and sulphate salts is leached for four days at 90°C. Oxidation of the uranium and residual sulphide minerals is accomplished by sparging with 99.5% oxygen. The mill tailings are disposed of directly (9).

UTS-4

The tailings sample for UTS-4 was provided by the Extractive Metallurgy Laboratory of CANMET which had previously prepared it from ore donated by, and using the recovery process of, Eldor Mines (formerly Gulf Minerals Canada Limited) at Rabbit Lake, Saskatchewan. The host rock for the uranium mineralization is a siliceous dolomite that has been highly altered and fractured. The ore consists of a high-grade zone of such mineralization in the centre of a brecciated zone, grading to low grade in the lesser brecciated perimeter (10). The comminuted ore is leached with sulphuric acid and sodium chlorate in

seven stages whereby pH and temperature are changed from 2.5-2.8 and 30-34°C to 1.6-1.7 and 48-52°C, respectively. The leach retention time is approximately eight hours. The mill tailings are neutralized with lime before disposal (10).

The four uranium tailings samples were dried at 80°C and dry-ground in November-December 1982 to pass a 104-μm sieve. The powdered tailings samples were each tumbled in one lot in a 570-L conical blender for 15 h and bottled. Approximately 340 kg of each of UTS-1, UTS-2 and UTS-3 were bottled in 200-g units. For UTS-4, approximately 134 kg were bottled in 100-g units. The four tailings samples were found to be sufficiently homogeneous for uranium by a neutron activation analytical technique and for total iron by an acid dissolution-atomic absorption spectrometric procedure to qualify as reference materials. Results of the confirmation of the homogeneity of UTS-1, UTS-2, UTS-3 and UTS-4 are reported in Appendix A.

The particle size analysis of the tailings samples is given in Table 1.

Table 1 – Particle size analysis of uranium tailings samples (wet screen)

Size of fraction (μm)	UTS-1	UTS-2	UTS-3	UTS-4
	mass %			
– + 104	0.1	0.03	<0.01	<0.01
–104 + 74	47.8	40.9	54.8	19.7
– 74 + 55	17.6	15.6	18.5	13.1
– 55 + 37	7.0	6.9	6.3	5.7
– 37	27.5	36.6	20.4	61.5

The concentration of the elements/constituents and the activity of the selected radioisotopes in the uranium tailings samples were determined by interlaboratory programs involving eighteen and eight laboratories, respectively. All participants except one for each program performed the requested analytical work under contract to CANMET with arrangements made by Supply and Services Canada (Tables A-1 and B-1).

ESTIMATION OF CONSENSUS VALUES AND 95% CONFIDENCE LIMITS

The estimation of the consensus value and related statistical parameters by the following procedure is performed only after outlying values are removed. The criteria for removal is peculiar to each interlaboratory program and as such is discussed in Parts A and B.

A one-way analysis of variance technique was used to estimate the consensus value and its variance. This approach considers the results of the described certification program to be only one sampling out of a universal set of results. The analytical data were assumed to fit the model (11)

$$x_{ij} = \mu + y_i + e_{ij}$$

where x_{ij} = the j^{th} result in set i,

μ = the true consensus value,

y_i = the discrepancy between the mean of the results in set i ($\bar{x}_{i.}$) and μ, and

e_{ij} = the discrepancy between x_{ij} and $\bar{x}_{i.}$.

It is assumed that both y_i and e_{ij} are normally distributed with means of zero and variances of ω and σ^2, respectively. The significance of ω^2 is detected by comparing the ratio of between-set mean squares and within-set mean squares with the F statistic at the 95% confidence level and with the appropriate degrees of freedom.

The consensus value of the assumed model is estimated by the overall mean $\bar{x}_{..}$:

$$\bar{x}_{..} = \sum_{i}^{k} \sum_{j}^{n_i} x_{ij} \Big/ \sum_{i}^{k} n_i$$

where n_i = the number of results in set i and

k = the number of sets.

The value of σ^2 is estimated by s_1^2 which is given by

$$s_1^2 = \sum_i^k \sum_j^{n_i} (x_{ij} - \bar{x}_{i.})^2 \Big/ \sum_i^k n_i - k$$

The value of ω^2 is estimated by

$$\omega^2 = (s_2^2 - s_1^2) \Big/ \frac{1}{k-1} \left(\sum_i^k n_i - \sum_i^k n_i^2 / \sum_i^k n_i \right)$$

where

$$s_2^2 = \sum_i^k n_i (\bar{x}_{i.} - \bar{x}_{..})^2 \Big/ k - 1$$

The variance of the overall mean is given by

$$v[\bar{x}_{..}] = \left(\sum_i^k n_i^2 / (\sum_i^k n_i)^2 \right) \omega^2 + \left(\sum_i^k 1 / \sum_i^k n_i \right) \sigma^2$$

and the 95% confidence limits for $\bar{x}_{..}$ are

$$\bar{x}_{..} \pm t_{0.975,\,(k-1)} \sqrt{v[\bar{x}_{..}]}$$

It should be noted that 95% confidence limits denote that if the interlaboratory program were performed 100 times, it would be expected that the overall mean in 95 would fall within the prescribed limits.

The average within-set standard deviation, σ_A, is a measure of the average within-bottle precision as determined by the analytical methods used. The implication exists, therefore, that a laboratory using a method of average or better reproducibility should obtain individual results for a given certified element with a repeatability that is at least comparable to the reported value of σ_A.

The consensus values for the elements/constituents and the radioactive isotopes are reported in Tables 2 and 3 respectively and are given recommended value status.

Table 2 – Recommended values for elements/constituents

Element/ constituent	UTS-1	UTS-2	UTS-3	UTS-4
	mass %			
Fe (total)	4.87	3.20	3.25	2.62
Ti	0.54	0.18	0.23	0.24
Al	6.24	2.71	5.80	6.29
Ca	5.24	0.42	4.03	1.75
S (total)	1.00	3.23	0.23	1.80
sulphate	2.64	0.84		5.21
	µg/g			
barium	324	464	212	65
uranium	49	56	513	1010
thorium	138	174	10.0	15.4
arsenic				38

Table 3 – Recommended activity values for radioactive isotopes

Isotope	UTS-1	UTS-2	UTS-3	UTS-4
	Bq/g			
Th-230	3.6	4.4	11.3	22.9
Ra-226	3.67	5.6	13.3	38.6
Pb-210	3.25	4.55	12.6	32.4
Po-210	3.1	4.4	11.8	30.8
Th-232	0.68	0.88		
Ra-228	0.68	1.0		
Th-228	0.71	0.92		

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CONFIRMATION OF HOMOGENEITY

CONFIRMATION OF HOMOGENEITY

The homogeneity of the uranium tailings samples was confirmed by Chemex Laboratories Ltd., North Vancouver, British Columbia, (Contract 15SQ.23440-2-9146) by analyzing for uranium and total iron in three separate subsamples for each of 19 bottles selected from a stock of 1655 bottles for UTS-1, UTS-2 and UTS-3 and of 1280 bottles for UTS-4. The stock of UTS-1 to UTS-3 was divided into 18 lots of 87 bottles and a 19th lot of 89 bottles; the stock of UTS-4 was divided into 18 lots of 67 bottles and a 19th lot of 74 bottles. In each case, the code number of the first bottle was selected at random out of the first lot. The code numbers of the remaining 18 bottles were given by the code number of the preceding bottle plus the number of bottles in each of the first 18 lots.

Uranium was determined by an instrumental neutron activation analytical technique. Total iron was determined by atomic absorption spectrometry after multi-acid sample decomposition. The results are shown in Tables 4 to 7.

A one-way analysis of variance techniques was used to assess the homogeneity (11). Herein, the ratio of the between-bottle to within-bottle mean square is compared with the F statistic at the 95% level of probability. There was no statistical evidence for bottle-to-bottle inhomogeneity with respect to uranium in all four tailings samples or with respect to iron for UTS-1 and UTS-2.

The iron results for UTS-3 and UTS-4 did indicate some bottle-to-bottle inhomogeneity. This however does not imply that the inhomogeneity is physically significant particularly in view of the contradictory results observed for uranium. Moreover, a detected inhomogeneity, statistical, physical or both, does not necessarily disqualify a candidate reference material from its intended use provided that its magnitude is acceptable in comparison with the overall uncertainty in the recommended value for the element of interest. The between-bottle standard deviation for iron in UTS-3 and UTS-4 was calculated to be 0.026 and 0.015% for the overall means of 3.12 and 2.60% Fe, respectively. The detected inhomogeneity therefore gives rise to relative uncertainties of 0.83 and 0.58% for UTS-3 and UTS-4, values which CCRMP concluded would be acceptable in comparison with the between-laboratory relative uncertainty expected from the results of the interlaboratory program. The latter can be calculated to be 0.089 and 0.073% Fe for UTS-3 and UTS-4, i.e., the relative certainty is 2.74 and 2.79%, respectively, thereby demonstrating UTS-3 and UTS-4 to be sufficiently homogeneous for use as reference materials.

Table 4a – Confirmation of homogeneity of UTS-1 for uranium

Bottle No.	Uranium (μg/g) Individual			Mean
40	54	53	48	51.7
127	47	49	51	49.0
214	51	49	50	50.0
301	53	52	50	51.7
388	49	49	48	48.7
475	52	49	51	50.7
565	50	50	54	51.3
649	51	48	48	49.0
736	50	49	51	50.0
823	49	49	47	48.3
910	52	49	49	50.0
997	52	48	48	49.3
1084	52	49	45	48.7
1171	48	48	49	48.3
1258	48	49	52	49.7
1345	51	51	49	50.3
1432	50	50	53	51.0
1519	49	47	51	49.0
1606	51	47	49	49.0

Overall mean = 49.8

Table 4b – Confirmation of homogeneity of UTS-1 for total iron

Bottle No.	Total iron (%) Individual			Mean
40	4.75	4.74	4.83	4.77
127	4.93	4.75	4.88	4.85
214	4.80	4.80	4.76	4.79
301	4.83	4.82	4.82	4.82
388	4.81	4.75	4.73	4.76
475	4.77	4.76	4.76	4.76
565	4.83	4.83	4.77	4.81
649	4.78	4.85	4.82	4.82
736	4.86	4.81	4.76	4.81
823	4.77	4.83	4.81	4.80
910	4.84	4.86	4.88	4.86
997	4.83	4.82	4.82	4.82
1084	4.79	4.90	4.90	4.86
1171	4.75	4.77	4.89	4.80
1258	4.86	4.81	4.77	4.81
1345	4.82	4.80	4.81	4.81
1432	4.78	4.77	4.78	4.78
1519	4.80	4.78	4.88	4.82
1606	4.83	4.81	4.87	4.84

Overall mean = 4.81

Analysis of variance table for uranium

Source of variation	Degrees of freedom	Mean square
Between bottles	18	3.595
Within bottles	38	3.614
Total	56	
Calculated F statistic =	0.995	
F.95 (18,38) =	1.883	

Null hypothesis of no difference between bottles is accepted for uranium

Analysis of variance table for total iron

Source of variation	Degrees of freedom	Mean square
Between bottles	18	2.645×10^{-3}
Within bottles	38	1.861×10^{-3}
Total	56	
Calculated F statistic =	1.421	
F.95 (18,38) =	1.883	

Null hypothesis of no difference between bottles is accepted for iron

Table 5a – Confirmation of homogeneity of UTS-2 for uranium

Bottle No.	Uranium (μg/g) Individual			Mean
72	53	54	55	54.0
159	52	53	56	53.7
246	50	57	58	55.0
333	55	56	54	55.0
420	55	55	55	55.0
507	57	57	54	56.0
594	56	57	55	56.0
681	55	56	55	55.3
768	54	55	57	55.3
855	55	55	57	55.7
942	57	57	55	56.3
1029	55	56	56	55.7
1116	55	54	55	54.7
1203	54	56	54	54.7
1290	54	55	57	55.3
1377	56	56	55	55.7
1464	54	57	56	55.7
1551	56	55	57	56.0
1638	56	53	54	54.3

Overall mean = 55.2

Table 5b – Confirmation of homogeneity of UTS-2 for total iron

Bottle No.	Total iron (%) Individual			Mean
72	3.05	3.05	3.14	3.08
159	3.08	3.07	3.15	3.10
246	3.09	3.12	3.05	3.09
333	3.07	3.09	3.14	3.10
420	3.11	3.08	3.01	3.07
507	3.04	3.05	3.08	3.06
594	3.09	3.09	3.05	3.08
681	3.03	3.13	3.05	3.07
768	3.04	3.08	3.09	3.07
855	3.09	3.03	3.04	3.05
942	3.10	3.13	3.19	3.14
1029	3.15	3.15	3.12	3.14
1116	3.11	3.10	3.04	3.08
1203	3.10	3.12	3.11	3.11
1290	3.05	3.16	3.07	3.09
1377	3.12	3.07	3.14	3.11
1464	3.06	3.07	3.10	3.08
1551	3.08	3.13	3.07	3.09
1638	3.07	3.04	3.06	3.06

Overall mean = 3.09

Analysis of variance table for uranium

Source of variation	Degrees of freedom	Mean square
Between bottles	18	1.558
Within bottles	38	2.368
Total	56	
Calculated F statistic =	0.658	
F.95 (18,38) =	1.883	

Null hypothesis of no difference between bottles is accepted for uranium

Analysis of variance table for total iron

Source of variation	Degrees of freedom	Mean square
Between bottles	18	1.888×10^{-3}
Within bottles	38	1.349×10^{-3}
Total	56	
Calculated F statistic =	1.400	
F.95 (18,38) =	1.883	

Null hypothesis of no difference between bottles is accepted for iron

Table 6a – Confirmation of homogeneity of UTS-3 for uranium

Bottle No.	Uranium (%) Individual			Mean
43	.0513	.0515	.0519	.0516
130	.0517	.0522	.0527	.0522
217	.0513	.0500	.0501	.0505
304	.0521	.0485	.0494	.0500
391	.0507	.0521	.0520	.0516
478	.0506	.0513	.0518	.0512
565	.0515	.0521	.0497	.0511
652	.0516	.0521	.0513	.0517
739	.0517	.0510	.0524	.0517
826	.0517	.0502	.0497	.0505
913	.0511	.0518	.0515	.0515
1000	.0505	.0518	.0507	.0510
1087	.0508	.0504	.0518	.0510
1174	.0517	.0510	.0511	.0513
1261	.0518	.0514	.0512	.0515
1348	.0531	.0513	.0510	.0518
1435	.0513	.0522	.0521	.0519
1522	.0513	.0497	.0497	.0502
1609	.0517	.0525	.0518	.0520

Overall mean = .0513

Table 6b – Confirmation of homogeneity of UTS-3 for total iron

Bottle No.	Total iron (%) Individual			Mean
43	3.15	3.17	3.18	3.17
130	3.20	3.15	3.11	3.15
217	3.16	3.10	3.13	3.13
304	3.07	3.10	3.11	3.09
391	3.13	3.07	3.14	3.11
478	3.10	3.05	3.13	3.09
565	3.06	3.05	3.08	3.06
652	3.09	3.08	3.06	3.08
739	3.20	3.20	3.13	3.18
826	3.15	3.11	3.15	3.14
913	3.13	3.15	3.15	3.14
1000	3.19	3.15	3.10	3.15
1087	3.06	3.16	3.14	3.12
1174	3.09	3.11	3.14	3.11
1261	3.10	3.06	3.10	3.09
1348	3.12	3.19	3.16	3.16
1435	3.14	3.07	3.10	3.10
1522	3.13	3.15	3.13	3.14
1609	3.13	3.10	3.10	3.11

Overall mean = 3.12

Analysis of variance table for uranium

Source of variation	Degrees of freedom	Mean square
Between bottles	18	1.117×10^{-6}
Within bottles	38	6.628×10^{-7}
Total	56	
Calculated F statistic =	1.685	
F.95 (18,38) =	1.883	

Null hypothesis of no difference between bottles is accepted for uranium

Analysis of variance table for total iron

Source of variation	Degrees of freedom	Mean square
Between bottles	18	2.960×10^{-3}
Within bottles	38	9.649×10^{-4}
Total	56	
Calculated F statistic =	3.068	
F.95 (18,38) =	1.883	

Null hypothesis of no difference between bottles is rejected for iron

Table 7a – Confirmation of homogeneity of UTS-4 for uranium

Bottle No.	Uranium (%) Individual			Mean
51	.1079	.1076	.1057	.1071
119	.1057	.1069	.1056	.1061
187	.1068	.1069	.1076	.1071
255	.1063	.1055	.1054	.1057
323	.1019	.1051	.1063	.1044
391	.1064	.1065	.1048	.1059
459	.1060	.1060	.1077	.1066
527	.1092	.1065	.1078	.1078
595	.1060	.1082	.1065	.1069
663	.1060	.1075	.1036	.1057
731	.1076	.1064	.1066	.1069
799	.1067	.1045	.1066	.1059
867	.1045	.1060	.1063	.1056
935	.1064	.1066	.1068	.1066
1003	.1063	.1072	.1058	.1064
1071	.1075	.1051	.1078	.1068
1139	.1070	.1069	.1065	.1068
1207	.1071	.1056	.1065	.1064
1275	.1062	.1059	.1043	.1055

Overall mean = .1063

Table 7b – Confirmation of homogeneity of UTS-4 for total iron

Bottle No.	Total iron (%) Individual			Mean
51	2 64	2 61	2 63	2 63
119	2 63	2 61	2 58	2 61
187	2 62	2 62	2 60	2 61
255	2 57	2 59	2 60	2 59
323	2 58	2 59	2 55	2 57
391	2 58	2 60	2 58	2 59
459	2 56	2 55	2 59	2 57
527	2 58	2 57	2 59	2 58
595	2 55	2 58	2 60	2 58
663	2 59	2 61	2 62	2 61
731	2 59	2 61	2 63	2 61
799	2 63	2 61	2 61	2 62
867	2 64	2 62	2 60	2 62
935	2 65	2 60	2 58	2 61
1003	2 59	2 60	2 57	2 59
1071	2 59	2 61	2 62	2 61
1139	2 61	2 63	2 63	2 62
1207	2 61	2 58	2 58	2 59
1275	2 61	2 61	2 64	2 62

Overall mean = 2 60

Analysis of variance table for uranium

Source of variation	Degrees of freedom	Mean square
Between bottles	18	1.772×10^{-6}
Within bottles	38	1.250×10^{-6}
Total	56	
Calculated F statistic =	1.418	
F.95 (18,38) =	1.883	

Null hypothesis of no difference between bottles is accepted for uranium

Analysis of variance table for total iron

Source of variation	Degrees of freedom	Mean square
Between bottles	18	1.040×10^{-3}
Within bottles	38	3.491×10^{-4}
Total	56	
Calculated F statistic =	2 980	
F.95 (18,38) =	1 883	

Null hypothesis of no difference between bottles is rejected for iron

PART A

RECOMMENDED VALUES FOR ELEMENTS/CONSTITUENTS IN UTS-1 to UTS-4

INTERLABORATORY PROGRAM

Participating laboratories in the interlaboratory program for the elements/constituents samples are listed in alphabetical order in Table A-1. Each was assigned a code number which bears no relationship to its alphabetical order.

Each laboratory was requested to contribute five replicate results for as many as possible of total iron, titanium, aluminum, calcium, barium, uranium, thorium, total sulphur and sulphate on one bottle of each of UTS-1, UTS-2, UTS-3 and UTS-4 by methods of its choice and to report the results on an "as is" basis. In addition, results for nickel and arsenic were requested for UTS-4. Some laboratories contributed results by more than one method: herein each set was considered statistically independent.

The results of the confirmation of the homogeneity of the tailings samples were included in the interlaboratory program. However, to avoid any biasing of the statistics, only five results, chosen at random out of the 57 available, were used in subsequent calculations. Analytical information is presented in Tables A-2 to A-5. Methodological information is presented in Table A-6 and pertains to all samples with the exception of nickel and arsenic.

STATISTICAL TREATMENT OF ANALYTICAL RESULTS

The consensus values and related statistical parameters were calculated as described above after outlying results were removed. Any sets of results obviously suspect for methodological reasons were rejected. Sets having unusually high variance were examined and any individual outlying results were deleted. Also, the sets of results whose means differed by more than twice the overall standard deviation from the initially calculated mean value were not used in subsequent computations to avoid biasing the statistics. All results that were rejected are identified in Tables A-2 to A-5.

The consensus values and related statistical parameters are summarized in Table A-7.

DISCUSSION

Table A-6 is a summary of a methodological classification of accepted analytical results where there is a clear-out distinction between types of methods in decomposition, separations and determination steps. No attempt was made to detect a statistically significant difference between the overall means of the more common methods for any element or constituent.

The consensus values of the interlaboratory program have been given recommended value status if there were at least nine sets of results and if the between-laboratory agreement was judged on the basis of chemical experience to be reasonable. The values for sulphate in UTS-3 and nickel in UTS-4 do not satisfy these criteria.

The between-laboratory agreement is, in many cases, not as good as is found in general in the interlaboratory programs of CCRMP, but it is, nevertheless, still acceptable to use these tailings samples as reference materials. Previous experience in CCRMP is that magnitude of the uncertainty in a consensus value has little effect on the actual estimation of the consensus value if the number of sets of results is sufficiently large.

Approximate Values

Laboratory 1 provided approximate values for the concentration of SiO_2, Na_2O and K_2O; they are reported in Table A-7.

Sulphur Control

The ratio of sulphate sulphur to total sulphur is an important parameter with respect to the potential environmental hazard posed by uranium tailings as a result of acid generation due to oxidation processes. The values of the ratios for UTS-1 and UTS-4 indicate that most of the sulphur is already present as sulphate and therefore should pose few problems with respect to acid generation. The low total sulphur content of UTS-3 is of course due to prior pyrite mineral separation by flotation. UTS-2 on the other hand contains appreciable amounts of oxidizable sulphur that can lead to deleterious acid generation, a phenomenon which is now being studied in detail in the Elliot Lake area.

Table A-1 – Contributing laboratories

Acme Analytical Laboratories Ltd.
Vancouver, British Columbia
(Dean Toye)
Contract 15SQ.23440-2-9144-1

Assayers (Ontario) Limited
Toronto, Ontario
(J. van Engelen)
Contract 26SQ.23400-5-9101-2

Atomic Energy of Canada Limited,
Radiochemical Company
Kanata, Ontario
(B.F. Raby)

Barringer Magenta Limited
Calgary, Alberta
(C.D. Read)
Contract 15SQ.23440-2-9144-2

Barringer Magenta Limited
Rexdale, Ontario
(R.E. Lett)
Contract 15SQ.23440-2-9144-3

Becquerel Laboratories Inc.
Mississauga, Ontario
(R. Robertson)
Contract 15SQ.23440-3-9116-1

Bondar-Clegg and Company Limited
Ottawa, Ontario
(P. Haulena)
Contract 15SQ.23440-3-9144-4

Bondar-Clegg and Company Limited
North Vancouver, British Columbia
(K.E. Rogers)
Contract 15SQ.23440-3-9144-5

CAN TEST Limited
Vancouver, British Columbia
(R.S. Jornitz)
Contract 15SQ.23440-3-9144-11

Chemex Laboratories Limited
North Vancouver, British Columbia
(R.D. Morse)
Contract 15SQ.23440-3-9144-6

Eco-Tech Laboratories Limited
Kamloops, British Columbia
(F.J. Pezzotti)
Contract 26SQ.23440-3-9101-1

Kamloops Research and Assay Laboratory Limited
Kamloops, British Columbia
(D.A. Blundell)
Contract 15SQ.23440-3-9144-7

Lakefield Research of Canada Limited
Lakefield, Ontario
(A.E. Carr)
Contract 26SQ.23440-3-9101-4

Materials Research Laboratory Limited
Nepean, Ontario
(S.K. Singh)
Contract 15SQ.23440-3-9144-8

Metriclab (1980) Inc.
Ste-Marthe-sur-le-lac, Quebec
(H. Blais)
Contract 26SQ.23440-3-9101-3

Saskatchewan Research Council
Saskatoon, Saskatchewan
(G. Smithson)
Contract SQ.23440-3-9116-2

Technical Service Laboratories
Mississauga, Ontario
(A.H. Debnam)
Contract 15SQ.23440-3-9144-9

X-Ray Assay Laboratories Limited
Don Mills, Ontario
(J.H. Opdebeeck)
Contract 15SQ.23440-3-9144-10

Table A-2a – Laboratory results, means and standard deviations for total iron and titanium in UTS-1

FE(TOTAL)

						MEAN	S.D.
LAB- 1 (PLASMA)	4.79	4.86	4.92	4.83	4.67	4.8140	.0934
LAB- 2 (AA)	4.74	4.83	4.74	4.83	4.83	4.7940	.0493
LAB- 3 (AA)	4.83	4.74	4.74	4.83	4.83	4.7940	.0493
LAB- 3 (PLASMA)	4.81	4.75	4.83	4.92	4.83	4.8280	.0610
LAB- 4 (AA)	4.80	4.84	4.72	4.80	4.80	4.7920	.0438
LAB- 5 (AA)	5.10	5.10	5.00	5.05	5.10	5.0700	.0447
LAB- 6 (TITR)	4.81	4.78	4.81	4.76	4.80	4.7920	.0217
LAB- 6 (AA)	4.75	4.74	4.83	4.81	4.87	4.8000	.0548
LAB- 7 (AA)	5.02	4.97	5.12	5.13	5.03	5.0540	.0688
*LAB- 8 (AA)	5.58	5.72	5.69	5.78	5.79	5.7120	.0847
LAB- 9 (PLASMA)	4.85	4.87	4.91	4.94	4.83	4.8800	.0447
LAB-10 (XRF)	4.995	4.985	4.955	4.935	4.965	4.9670	.0239
LAB-11 (PLASMA)	4.94	4.83	4.85	4.85	4.88	4.8700	.0430

TITANIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	0.57	0.56	0.55	0.58	0.55	.5620	.0130
LAB- 2 (AA)	0.58	0.58	0.58	0.58	0.58	.5800	0.0000
LAB- 3 (AA)	0.58	0.63	0.58	0.58	0.58	.5900	.0224
LAB- 3 (PLASMA)	0.535	0.555	0.552	0.544	0.561	.5494	.0101
*LAB- 4 (COLOR)	0.33	0.33	0.32	0.33	0.34	.3300	.0071
LAB- 5 (COLOR)	0.54	0.54	0.51	0.48	0.51	.5160	.0251
LAB- 6 (COLOR)	0.529	0.514	0.521	0.517	0.525	.5212	.0060
LAB- 7 (AA)	0.48	0.46	0.45	0.46	0.45	.4600	.0122
*LAB- 8 (AA)	0.33	0.33	0.32	0.32	0.32	.3240	.0055
LAB- 9 (PLASMA)	0.52	0.55	0.54	0.55	0.52	.5360	.0152
LAB-10 (XRF)	0.525	0.535	0.525	0.520	0.535	.5280	.0067
LAB-11 (PLASMA)	0.585	0.582	0.577	0.586	0.588	.5836	.0043

**Outliers, not used for computations*

Table A-2b – Laboratory results, means and standard deviations for aluminum and calcium in UTS-1

ALUMINUM

						MEAN	S.D.
LAB- 1 (PLASMA)	6.35	6.19	6.24	6.30	6.30	6.2760	.0619
LAB- 2 (AA)	6.162	6.293	6.293	6.192	6.293	6.2466	.0644
LAB- 3 (AA)	6.293	5.895	5.859	5.927	5.996	5.9940	.1746
LAB- 3 (PLASMA)	6.35	6.35	6.30	6.35	6.46	6.3620	.0589
LAB- 4 (AA)	5.92	6.08	6.00	6.12	5.88	6.0000	.1020
LAB- 5 (AA)	6.45	6.45	6.40	6.51	6.40	6.4420	.0455
LAB- 6 (AA)	6.36	6.37	6.34	6.39	6.38	6.3680	.0192
LAB- 7 (AA)	6.07	5.95	6.20	6.07	6.10	6.0760	.0893
LAB- 8 (AA)	6.34	6.43	6.23	6.26	6.35	6.3220	.0792
LAB- 9 (PLASMA)	6.32	6.34	6.32	6.31	6.30	6.3180	.0148
LAB-10 (XRF)	6.25	6.26	6.24	6.21	6.255	6.2430	.0199
LAB-11 (PLASMA)	6.28	6.22	6.27	6.25	6.27	6.2580	.0239

CALCIUM

						MEAN	S.D.
*LAB- 1 (PLASMA)	4.90	4.82	4.73	4.70	4.80	4.7900	.0787
LAB- 2 (AA)	5.32	5.32	5.32	5.32	5.32	5.3200	.0000
LAB- 3 (AA)	5.22	5.32	5.32	5.32	5.32	5.3000	.0447
LAB- 3 (PLASMA)	5.30	5.28	5.27	5.22	5.17	5.2480	.0526
LAB- 4 (AA)	5.00	5.04	5.04	5.12	5.08	5.0560	.0456
LAB- 5 (AA)	5.14	5.18	5.22	5.14	5.14	5.1640	.0358
LAB- 6 (AA)	5.17	5.11	5.10	5.16	5.12	5.1320	.0311
LAB- 7 (AA)	5.36	5.39	5.36	5.46	5.54	5.4220	.0776
LAB- 8 (AA)	5.21	5.27	5.27	5.19	5.08	5.2040	.0780
LAB- 9 (PLASMA)	5.27	5.36	5.33	5.30	5.30	5.3120	.0342
LAB-10 (XRF)	5.225	5.235	5.205	5.19	5.24	5.2190	.0210
LAB-11 (PLASMA)	5.26	5.21	5.25	5.25	5.20	5.2340	.0270

Outliers, not used for computations

Table A-2c – Laboratory results, means and standard deviations for barium and uranium in UTS-1

BARIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	324.0	321.0	325.0	324.0	324.0	323.60	1.5166
LAB- 4 (XRF)	360.	400.	340.	400.	370.	374.00	26.0768
LAB- 5 (XRF)	370.0	390.0	400.0	390.0	390.0	388.00	10.9545
LAB- 6 (AE)	370.0	370.0	370.0	370.0	370.0	370.00	0.0000
LAB- 7 (AA)	330.	390.	360.	360.	300.	348.00	34.2053
*LAB- 8 (AA)	485.0	483.0	488.0	490.0	491.0	487.40	3.3615
LAB- 9 (PLASMA)	296.0	292.0	297.0	296.0	294.0	295.00	2.0000
LAB-10 (XRF)	350.	350.	360.	350.	350.	352.00	4.4721
LAB-11 (PLASMA)	342.	343.	342.	345.	349.	344.20	2.9496
LAB-15 (XRF)	294.	314.	333.	314.	282.	307.40	19.7939
LAB-17 (AA)	226.	242.	248.	281.	237.	246.80	20.7533
LAB-18 (XRF)	220.	220.	200.	230.	220.	218.00	10.9545

URANIUM

						MEAN	S.D.
*LAB- 1 (PLASMA)	78.0	74.0	82.0	80.0	77.0	78.20	3.0332
LAB- 2 (FLUOR)	55.0	50.0	45.0	50.0	50.0	50.00	3.5355
LAB- 2 (NAA)	48.9	48.4	50.8	48.6	45.2	48.38	2.0179
LAB- 3 (FLUOR)	65.0	60.0	65.0	50.0	55.0	59.00	6.5192
LAB- 3 (NAA)	51.3	53.7	49.5	51.2	48.8	50.90	1.9013
LAB- 4 (FLUOR)	53.0	54.0	50.0	46.0	47.0	50.00	3.5355
LAB- 5 (FLUOR)	31.0	29.0	28.0	30.0	29.0	29.40	1.1402
LAB- 6 (NAA)	50.0	53.0	49.0	50.0	52.0	50.80	1.6432
LAB- 6 (NAA)	54.	53.	48.	47.	49.	50.20	3.1145
LAB- 7 (FLUOR)	30.	41.	50.	31.	44.	39.20	8.5849
LAB- 8 (FLUOR)	52.3	53.6	53.9	54.6	48.0	52.48	2.6395
LAB- 9 (FLUOR)	54.0	63.0	65.0	59.0	60.0	60.20	4.2071
LAB-10 (NAA)	51.7	49.2	49.3	51.5	50.7	50.48	1.1841
*LAB-11 (FLUOR)	23.0	25.0	24.0	22.0	25.0	23.80	1.3038

**Outliers, not used for computations*

Table A-2d – Laboratory results, means and standard deviations for thorium and total sulphur in UTS-1

THORIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	165.0	153.0	151.0	155.0	150.0	154.80	6.0166
LAB- 2 (NAA)	140.0	140.0	140.0	130.0	130.0	136.00	5.4772
LAB- 3 (NAA)	130.0	140.0	130.0	140.0	130.0	134.00	5.4772
LAB- 3 (PLASMA)	136.0	142.0	139.0	136.0	141.0	138.80	2.7749
LAB- 4 (XRF)	145.	143.	135.	150.	136.	141.80	6.3008
LAB- 5 (XRF)	137.0	144.0	139.0	140.0	143.0	140.60	2.8810
LAB- 6 (NAA)	170.0	170.0	160.0	170.0	150.0	164.00	8.9443
*LAB- 7 (COLOR)	108.	94.	99.	97.	102.	100.00	5.3385
LAB- 8 (COLOR)	138.0	143.0	126.0	123.0	126.0	131.20	8.7579
LAB- 9 (RADIO)	119.0	145.0	122.0	100.0	105.0	118.20	17.5983
LAB-10 (NAA)	135.	131.	138.	139.	138.	136.20	3.2711
LAB-11 (NAA)	126.	125.	130.	131.	125.	127.40	2.8810

S(TOTAL)

						MEAN	S.D.
LAB- 1 (TITR)	0.888	0.887	0.955	0.944	0.964	.9276	.0373
LAB- 2 (TITR)	0.9	0.9	0.9	0.9	1.0	.9200	.0447
LAB- 3 (TITR)	0.9	1.0	0.9	0.9	1.0	.9400	.0548
LAB- 4 (TITR)	1.04	1.07	1.03	1.16	1.14	1.0880	.0589
LAB- 5 (TITR)	0.94	0.96	0.95	0.96	0.97	.9560	.0114
LAB- 6 (GRAV)	1.004	1.010	0.999	0.999	0.993	1.0010	.0064
LAB- 7 (GRAV)	0.99	1.00	0.99	1.05	1.01	1.0080	.0249
LAB- 8 (COLOR)	0.94	0.95	0.99	1.02	1.00	.9800	.0339
LAB- 9 (TITR)	0.98	1.01	1.00	1.04	1.00	1.0060	.0219
*LAB-10 (XRF)	1.625	1.645	1.645	1.68	1.615	1.6420	.0249
LAB-11 (TITR)	1.20	1.15	1.15	1.17	1.17	1.1680	.0205

Outliers, not used for computations

Table A-2e – Laboratory results, means and standard deviations for sulphate in UTS-1

SULPHATE

						MEAN	S.D.
LAB- 1 (GRAV)	2.463	2.484	2.622	2.580	2.595	2.5488	.0708
LAB- 4 (TITR)	2.74	2.76	2.72	2.71	2.72	2.7300	.0200
*LAB- 5 (COMB)	0.88	0.93	0.87	0.91	0.91	.9000	.0245
LAB- 6 (GRAV)	2.695	2.683	2.721	2.715	2.728	2.7084	.0188
LAB- 7 (GRAV)	2.68	2.62	2.66	2.69	2.70	2.6700	.0316
*LAB- 8 (COLOR)	1.71	1.68	1.67	1.66	1.69	1.6820	.0192
LAB- 9 (GRAV)	2.56	2.50	2.47	2.49	2.51	2.5060	.0336
LAB-10 (GRAV)	2.61	2.52	2.38	2.635	2.57	2.5430	.1010
LAB-11 (GRAV)	2.65	2.70	2.64	2.65	2.64	2.6560	.0251
*LAB-15 (GRAV)	1.83	1.83	1.81	1.80	1.84	1.8220	.0164
LAB-16 (GRAV)	2.71	2.70	2.69	2.69	2.71	2.7000	.0100
*LAB-17 (COMB)	.262	.298	.300	.265	.290	.2830	.0182
LAB-18 (GRAV)	2.70	2.70	2.70	2.72	2.71	2.7060	.0089

**Outliers, not used for computations*

Table A-3a – Laboratory results, means and standard deviations for total iron and titanium in UTS-2

FE(TOTAL)

						MEAN	S.D.
LAB- 1 (PLASMA)	3.34	3.46	3.35	3.44	3.58	3.4340	.0974
LAB- 2 (AA)	3.04	3.13	3.13	3.13	3.13	3.1120	.0402
LAB- 3 (AA)	3.04	3.13	3.13	3.13	3.13	3.1120	.0402
LAB- 3 (PLASMA)	3.15	3.14	3.18	3.18	3.18	3.1660	.0195
LAB- 4 (AA)	3.24	3.24	3.12	3.16	3.16	3.1840	.0537
LAB- 5 (AA)	3.20	3.20	3.25	3.20	3.25	3.2200	.0274
LAB- 6 (TITR)	3.04	3.00	2.98	2.99	3.00	3.0020	.0228
LAB- 6 (AA)	3.05	3.05	3.14	3.04	3.06	3.0680	.0409
LAB- 7 (AA)	3.06	3.12	3.12	2.97	3.06	3.0660	.0615
LAB- 8 (AA)	3.65	3.56	3.60	3.49	3.57	3.5740	.0586
*LAB- 9 (PLASMA)	2.72	2.64	2.75	2.71	2.70	2.7040	.0404
LAB-10 (XRF)	3.055	3.10	3.085	3.075	3.04	3.0710	.0238
LAB-11 (PLASMA)	3.36	3.31	3.30	3.34	3.36	3.3340	.0279

TITANIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	0.22	0.22	0.21	0.22	0.21	.2160	.0055
LAB- 2 (AA)	0.14	0.14	0.14	0.14	0.14	.1400	.0000
LAB- 3 (AA)	0.14	0.14	0.14	0.14	0.14	.1400	.0000
LAB- 3 (PLASMA)	0.138	0.143	0.135	0.123	0.132	.1342	.0075
LAB- 4 (COLOR)	0.200	0.20	0.21	0.22	0.19	.2040	.0114
LAB- 5 (COLOR)	0.21	0.18	0.24	0.21	0.21	.2100	.0212
LAB- 6 (COLOR)	0.202	0.202	0.210	0.203	0.206	.2046	.0034
LAB- 7 (AA)	0.16	0.14	0.15	0.15	0.16	.1520	.0084
LAB- 8 (AA)	0.21	0.21	0.22	0.20	0.22	.2120	.0084
LAB- 9 (PLASMA)	0.22	0.21	0.21	0.20	0.22	.2120	.0084
LAB-10 (XRF)	0.22	0.22	0.215	0.22	0.22	.2190	.0022
LAB-11 (PLASMA)	0.152	0.157	0.152	0.147	0.152	.1520	.0035

*Outliers, not used for computations

Table A-3b – Laboratory results, means and standard deviations for aluminum and calcium in UTS-2

ALUMINUM

						MEAN	S.D.
LAB- 1 (PLASMA)	2.67	2.83	2.65	2.73	2.70	2.7160	.0706
LAB- 2 (AA)	2.64	2.56	2.64	2.64	2.56	2.6080	.0438
LAB- 3 (AA)	2.60	2.56	2.53	2.57	2.57	2.5660	.0251
LAB- 3 (PLASMA)	2.80	2.75	2.72	2.77	2.76	2.7600	.0292
LAB- 4 (AA)	2.56	2.60	2.56	2.60	2.52	2.5680	.0335
LAB- 5 (AA)	2.65	2.70	2.73	2.75	2.70	2.7060	.0378
LAB- 6 (AA)	2.74	2.77	2.79	2.79	2.80	2.7780	.0239
LAB- 7 (AA)	2.74	2.87	2.87	2.67	2.80	2.7900	.0863
LAB- 8 (AA)	2.69	2.76	2.66	2.76	2.72	2.7180	.0438
LAB- 9 (PLASMA)	2.80	2.78	2.76	2.79	2.81	2.7880	.0192
LAB-10 (XRF)	2.90 *	2.69	2.685	2.68	2.715	2.7340	.0938
LAB-11 (PLASMA)	2.80	2.79	2.79	2.76	2.80	2.7880	.0164

CALCIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	0.36	0.36	0.35	0.37	0.36	.3600	.0071
LAB- 2 (AA)	0.47	0.47	0.47	0.47	0.47	.4700	.0000
LAB- 3 (AA)	0.47	0.47	0.47	0.47	0.47	.4700	.0000
LAB- 3 (PLASMA)	0.403	0.415	0.415	0.423	0.413	.4138	.0072
LAB- 4 (AA)	0.386	0.384	0.380	0.386	0.370	.3812	.0067
LAB- 5 (AA)	0.43	0.43	0.39	0.39	0.43	.4140	.0219
LAB- 6 (AA)	0.427	0.426	0.425	0.424	0.427	.4258	.0013
LAB- 7 (AA)	0.43	0.43	0.43	0.37	0.41	.4140	.0261
LAB- 8 (AA)	0.45	0.45	0.47	0.47	0.46	.4600	.0100
LAB- 9 (PLASMA)	0.35	0.32	0.34	0.35	0.33	.3380	.0130
LAB-10 (XRF)	0.44	0.44	0.44	0.44	0.44	.4400	.0000
LAB-11 (PLASMA)	0.43	0.43	0.42	0.43	0.42	.4260	.0055

**Outliers, not used for computations*

Table A-3c – Laboratory results, means and standard deviations for barium and uranium in UTS-2

BARIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	482.0	475.0	471.0	471.0	466.0	473.00	5.9582
LAB- 4 (XRF)	500.	480.	450.	540.	510.	496.00	33.6155
*LAB- 5 (XRF)	630.0	620.0	630.0	620.0	620.0	624.00	5.4772
LAB- 6 (AE)	490.0	490.0	490.0	490.0	500.0	492.00	4.4721
LAB- 7 (AA)	400.	410.	420.	420.	440.	418.00	14.8324
LAB- 8 (AA)	544.0	540.0	526.0	526.0	533.0	533.80	8.1363
LAB- 9 (PLASMA)	432.0	431.0	419.0	433.0	434.0	429.80	6.1400
LAB-10 (XRF)	520.	530.	530.	520.	510.	522.00	8.3666
LAB-11 (PLASMA)	510.	536.	537.	540.	533.	531.20	12.1120
LAB-15 (XRF)	400.	394.	398.	384.	400.	395.20	6.7231
LAB-17 (AA)	367.	336.	351.	390.	348.	358.40	20.8399
LAB-18 (XRF)	480.	460.	460.	460.	440.	460.00	14.1421

URANIUM

						MEAN	S.D.
*LAB- 1 (PLASMA)	67.0	68.0	66.0	71.0	65.0	67.40	2.3022
LAB- 2 (FLUOR)	55.0	60.0	55.0	55.0	55.0	56.00	2.2361
LAB- 2 (NAA)	56.0	54.0	52.3	55.3	59.6	55.44	2.7190
LAB- 3 (FLUOR)	60.0	50.0	60.0	60.0	50.0	56.00	5.4772
LAB- 3 (NAA)	53.7	53.8	53.3	54.5	54.0	53.86	.4393
LAB- 4 (FLUOR)	55.0	55.0	55.0	54.0	53.0	54.40	.8944
*LAB- 5 (FLUOR)	40.0	40.0	43.0	41.0	43.0	41.40	1.5166
LAB- 6 (NAA)	56.0	55.0	56.0	59.0	55.0	56.20	1.6432
LAB- 6 (NAA)	53.	54.	55.	53.	54.	53.80	.8367
*LAB- 7 (FLUOR)	56.	56.	73.	71.	67.	64.60	8.1425
LAB- 8 (FLUOR)	57.7	55.6	59.3	56.5	58.0	57.42	1.4237
LAB- 9 (FLUOR)	54.0	61.0	59.0	57.0	58.0	57.80	2.5884
LAB-10 (NAA)	56.0	56.9	55.4	56.5	56.6	56.28	.5891
*LAB-11 (FLUOR)	39.0	42.0	39.0	42.0	41.0	40.60	1.5166

Outliers, not used for computations

Table A-3d – Laboratory results, means and standard deviations for thorium and total sulphur in UTS-2

THORIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	189.0	182.0	182.0	197.0	191.0	188.20	6.3797
LAB- 2 (NAA)	170.0	170.0	180.0	170.0	170.0	172.00	4.4721
LAB- 3 (NAA)	170.0	170.0	160.0	160.0	180.0	168.00	8.3666
LAB- 3 (PLASMA)	179.0	179.0	179.0	187.0	173.0	179.40	4.9800
LAB- 4 (XRF)	177.	179.	185.	186.	188.	183.00	4.7434
LAB- 5 (XRF)	196.0	192.0	200.0	196.0	196.0	196.00	2.8284
LAB- 6 (NAA)	210.0	200.0	210.0	210.0	200.0	206.00	5.4772
LAB- 7 (COLOR)	132.	132.	123.	133.	126.	129.20	4.4385
LAB- 8 (COLOR)	171.0	171.0	157.0	160.0	164.0	164.60	6.3482
LAB- 9 (RADIO)	215.0	161.0	189.0	168.0	174.0	181.40	21.4313
LAB-10 (NAA)	166.	164.	171.	164.	161.	165.20	3.7014
LAB-11 (NAA)	159.	151.	162.	163.	157.	158.40	4.7749

S(TOTAL)

						MEAN	S.D.
LAB- 1 (TITR)	3.340	3.500	3.380	3.340	3.250	3.3620	.0907
LAB- 2 (TITR)	3.1	3.1	3.1	3.1	3.1	3.1000	.0000
LAB- 3 (TITR)	3.0	3.0	3.0	2.9	2.9	2.9600	.0548
LAB- 4 (TITR)	3.50	3.44	3.44	3.43	3.44	3.4500	.0283
LAB- 5 (TITR)	3.14	3.10	3.18	3.14	3.14	3.1400	.0283
LAB- 6 (GRAV)	3.364	3.308	3.362	3.255	3.362	3.3302	.0483
LAB- 7 (GRAV)	3.31	3.31	3.37	3.32	3.11	3.2840	.1004
LAB- 8 (COLOR)	3.43	3.41	3.50	3.31	3.36	3.4020	.0719
LAB- 9 (TITR)	3.17	3.26	3.31	3.29	3.21	3.2480	.0576
LAB-10 (XRF)	3.06	3.115	3.065	3.095	3.075	3.0820	.0228
LAB-11 (TITR)	3.17	3.15	3.20	3.25	3.15	3.1840	.0422

Table A-3e – Laboratory results, means and standard deviations for sulphate in UTS-2

SULPHATE

						MEAN	S.D.
*LAB- 1 (GRAV)	1.109	1.258	1.318	1.258	1.198	1.2282	.0790
LAB- 4 (TITR)	0.92	0.92	0.93	0.91	0.92	.9200	.0071
*LAB- 5 (COMB.)	0.31	0.36	0.31	0.35	0.35	.3360	.0241
LAB- 6 (GRAV)	0.895	0.911	0.902	0.925	0.912	.9090	.0113
LAB- 7 (GRAV)	0.94	0.95	0.96	0.94	0.94	.9460	.0089
LAB- 8 (COLOR)	0.88	0.89	0.92	0.86	0.86	.8820	.0249
LAB- 9 (GRAV)	0.68	0.70	0.75	0.71	0.69	.7060	.0270
LAB-10 (GRAV)	0.695	0.675	0.675	0.675	0.65	.6740	.0160
LAB-11 (GRAV)	0.91	0.90	0.91	0.92	0.90	.9080	.0084
LAB-15 (GRAV)	0.66	0.66	0.67	0.64	0.66	.6580	.0110
LAB-16 (GRAV)	0.892	0.901	0.900	0.901	0.896	.8980	.0039
*LAB-17 (COMB)	8.44	8.51	8.34	8.72	8.24	8.4500	.1822
LAB-18 (GRAV)	0.94	0.91	0.91	0.91	0.91	.9160	.0134

*Outliers, not used for computations

Table A-4a – Laboratory results, means and standard deviations for total iron and titanium in UTS-3

FE(TOTAL)

						MEAN	S.D.
LAB- 1 (PLASMA)	3.28	3.18	3.18	3.24	3.22	3.2200	.0424
LAB- 2 (AA)	3.31	3.31	3.31	3.31	3.31	3.3100	.0000
LAB- 3 (AA)	3.32	3.32	3.27	3.32	3.32	3.3100	.0224
LAB- 3 (PLASMA)	3.03	3.36	3.27	3.27	3.25	3.2360	.1228
LAB- 4 (AA)	3.08	3.16	3.20	3.12	3.20	3.1520	.0522
LAB- 5 (AA)	3.35	3.30	3.35	3.30	3.30	3.3200	.0274
LAB- 6 (TITR)	3.11	3.15	3.11	3.14	3.15	3.1320	.0205
LAB- 6 (AA)	3.15	3.17	3.18	3.10	3.10	3.1400	.0381
LAB- 7 (AA)	3.31	3.28	3.41	3.40	3.38	3.3560	.0577
*LAB- 8 (AA)	3.78	3.78	3.77	3.85	3.79	3.7940	.0321
LAB- 9 (PLASMA)	3.19	3.12	3.18	3.14	3.15	3.1560	.0288
LAB-10 (XRF)	3.425	3.405	3.38	3.355	3.39	3.3910	.0263
LAB-11 (PLASMA)	3.34	3.30	3.32	3.29	3.30	3.3100	.0200

TITANIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	0.24	0.25	0.24	0.24	0.24	.2420	.0045
LAB- 2 (AA)	0.22	0.22	0.22	0.22	0.22	.2200	.0000
LAB- 3 (AA)	0.22	0.22	0.22	0.22	0.22	.2200	.0000
LAB- 3 (PLASMA)	0.195	0.204	0.204	0.206	0.211	.2040	.0058
*LAB- 4 (COLOR)	0.33	0.33	0.33	0.34	0.34	.3340	.0055
LAB- 5 (COLOR)	0.27	0.24	0.24	0.21	0.21	.2340	.0251
LAB- 6 (COLOR)	0.247	0.243	0.247	0.247	0.251	.2470	.0028
LAB- 7 (AA)	0.17	0.16	0.17	0.18	0.17	.1700	.0071
LAB- 8 (AA)	0.23	0.24	0.24	0.24	0.24	.2380	.0045
LAB- 9 (PLASMA)	0.25	0.24	0.24	0.24	0.25	.2440	.0055
LAB-10 (XRF)	0.245	0.245	0.245	0.245	0.25	.2460	.0022
LAB-11 (PLASMA)	0.203	0.208	0.209	0.209	0.2100	.2078	.0028

*Outliers, not used for computations

Table A-4b – Laboratory results, means and standard deviations for aluminum and calcium in UTS-3

ALUMINUM

						MEAN	S.D.
LAB- 1 (PLASMA)	5.61	5.61	5.56	5.61	5.56	5.5900	.0274
LAB- 2 (AA)	5.700	5.800	5.800	5.800	5.70	5.7600	.0548
LAB- 3 (AA)	5.531	5.605	5.541	5.573	5.594	5.5688	.0323
LAB- 3 (PLASMA)	5.93	6.03	5.98	5.98	6.03	5.9900	.0418
LAB- 4 (AA)	5.60	5.64	5.56	5.52	5.68	5.6000	.0632
LAB- 5 (AA)	6.00	6.03	6.00	6.09	6.00	6.0240	.0391
LAB- 6 (AA)	6.02	6.04	5.91	5.98	6.02	5.9940	.0518
LAB- 7 (AA)	5.40	5.53	5.40	5.27	5.27	5.3740	.1088
LAB- 8 (AA)	5.90	5.88	5.93	5.94	5.98	5.9260	.0385
LAB- 9 (PLASMA)	5.93	5.85	5.88	5.86	5.90	5.8840	.0321
LAB-10 (XRF)	5.87	5.885	5.875	5.875	5.915	5.8840	.0182
LAB-11 (PLASMA)	6.03	5.95	5.99	5.97	5.99	5.9860	.0297

CALCIUM

						MEAN	S.D.
*LAB- 1 (PLASMA)	3.47	3.50	3.44	3.47	3.44	3.4640	.0251
LAB- 2 (AA)	4.09	4.00	4.09	4.00	4.18	4.0720	.0753
LAB- 3 (AA)	4.00	4.00	4.00	4.00	4.00	4.0000	0.0000
LAB- 3 (PLASMA)	3.92	3.99	3.81	3.99	3.97	3.9360	.0760
*LAB- 4 (AA)	3.44	3.52	3.44	3.56	3.56	3.5040	.0607
LAB- 5 (AA)	3.86	3.93	3.89	3.93	3.89	3.9000	.0300
LAB- 6 (AA)	4.07	4.02	4.05	4.02	4.08	4.0480	.0277
LAB- 7 (AA)	4.32	4.27	4.16	4.33	4.15	4.2460	.0862
LAB- 8 (AA)	4.08	4.09	4.02	4.12	4.09	4.0800	.0367
LAB- 9 (PLASMA)	3.97	3.92	4.01	3.96	3.95	3.9620	.0327
LAB-10 (XRF)	4.055	4.045	4.05	4.055	4.07	4.0550	.0094
LAB-11 (PLASMA)	4.00	3.95	3.97	3.96	3.99	3.9740	.0207

Outliers, not used for computations

Table A-4c – Laboratory results, means and standard deviations for barium and uranium in UTS-3

BARIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	191.0	202.0	191.0	196.0	191.0	194.20	4.8683
*LAB- 4 (XRF)	430.	430.	430.	420.	420.	426.00	5.4772
LAB- 5 (XRF)	230.0	230.0	240.0	240.0	220.0	232.00	8.3666
LAB- 6 (AE)	230.0	220.0	230.0	230.0	230.0	228.00	4.4721
LAB- 7 (AA)	250.	220.	210.	230.	210.	224.00	16.7332
LAB- 8 (AA)	273.0	279.0	264.0	286.0	280.0	276.40	8.3247
LAB- 9 (PLASMA)	204.0	178.0	186.0	167.0	185.0	184.00	13.5093
LAB-10 (XRF)	190.	180.	190.	180.	180.	184.00	5.4772
LAB-11 (PLASMA)	214.	214.	218.	212.	215.	214.60	2.1909
LAB-15 (XRF)	214.	218.	214.	216.	224.	217.20	4.1473
LAB-17 (AA)	219.	196.	218.	213.	185.	206.20	15.0233
LAB-18 (XRF)	160.	170.	170.	180.	180.	172.00	8.3666

URANIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	518.0	516.0	516.0	525.0	517.0	518.40	3.7815
LAB- 2 (FLUOR)	550.0	500.0	500.0	550.0	500.0	520.00	27.3861
LAB- 2 (NAA)	527.0	523.0	518.0	512.0	515.0	519.00	6.0415
LAB- 3 (FLUOR)	500.0	500.0	500.0	500.0	500.0	500.00	0.0000
LAB- 3 (NAA)	531.0	519.0	520.0	528.0	519.0	523.40	5.6833
LAB- 4 (FLUOR)	524.	528.	514.	516.	521.	520.60	5.7271
LAB- 5 (FLUOR)	485.0	515.0	485.0	485.0	490.0	492.00	13.0384
LAB- 6 (NAA)	516.0	518.0	521.0	492.0	500.0	509.40	12.6807
LAB- 6 (NAA)	513.	515.	519.	525.	518.	518.00	4.5826
LAB- 7 (FLUOR)	496.	500.	473.	480.	495.	488.80	11.6490
LAB- 8 (FLUOR)	546.0	569.0	548.0	574.0	543.0	556.00	14.3701
*LAB- 9 (FLUOR)	660.0	630.0	590.0	560.0	610.0	610.00	38.0789
LAB-10 (NAA)	551.	554.	550.	551.	550.	551.20	1.6432
LAB-11 (FLUOR)	450.	450.	460.	455.	450.	453.00	4.4721

Outliers, not used for computations

Table A-4d – Laboratory results, means and standard deviations for thorium and total sulphur in UTS-3

THORIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	15.0	13.0	13.0	13.0	13.0	13.400	.8944
LAB- 2 (NAA)	9.0	9.0	9.0	9.0	9.0	9.000	0.0000
LAB- 3 (NAA)	10.0	9.0	9.0	9.0	9.0	9.200	.4472
*LAB- 3 (PLASMA)	25.3	26.7	22.7	25.2	25.2	25.020	1.4446
*LAB- 4 (XRF)	131.	148.	136.	137.	131.	136.600	6.9498
LAB- 5 (XRF)	11.0	7.0	12.0	4.0	4.0	7.600	3.7815
LAB- 6 (NAA)	9.0	18.0	9.0	18.0	18.0	14.400	4.9295
*LAB- 7 (COLOR)	38.	34.	34.	35.	46.	37.400	5.0794
LAB- 8 (COLOR)	9.4	9.7	8.4	9.1	8.7	9.060	.5225
LAB-10 (NAA)	9.15	8.9	9.4	8.95	8.95	9.070	.2080
LAB-11 (NAA)	11.0	8.	12.0	11.0	11.0	10.600	1.5166
LAB-12 (NAA)	8.6	7.3	9.1	8.5	9.5	8.600	.8307
LAB-13 (NAA)	8.92	9.16	9.01	9.17	8.90	9.032	.1283
LAB-14 (NAA)	10.2	9.9	9.2	10.4	10.2	9.980	.4712

S(TOTAL)

						MEAN	S.D.
LAB- 1 (TITR)	0.192	0.206	0.217	0.217	0.225	.2114	.0128
LAB- 2 (TITR)	0.2	0.2	0.2	0.2	0.2	.2000	.0000
LAB- 3 (TITR)	0.2	0.2	0.2	0.2	0.2	.2000	.0000
LAB- 4 (TITR)	0.266	0.262	0.260	0.243	0.253	.2568	.0090
LAB- 5 (TITR)	0.22	0.22	0.21	0.23	0.22	.2200	.0071
LAB- 6 (GRAV)	0.209	0.209	0.220	0.220	0.226	.2168	.0075
LAB- 7 (GRAV)	0.21	0.22	0.27	0.24	0.24	.2360	.0230
LAB- 8 (COLOR)	0.22	0.25	0.26	0.26	0.25	.2480	.0164
LAB- 9 (TITR)	0.27	0.24	0.24	0.25	0.27	.2540	.0152
LAB-10 (XRF)	0.22	0.21	0.215	0.195	0.205	.2090	.0096
*LAB-11 (TITR)	0.33	0.30	0.31	0.27	0.30	.3020	.0217

Outliers, not used for computations

Table A-4e – Laboratory results, means and standard deviations for sulphate in UTS-3

SULPHATE

						MEAN	S.D.
*LAB- 1 (GRAV)	0.180	0.174	0.183	0.165	0.165	.1734	.0083
LAB- 4 (TITR)	0.021	0.022	0.012	0.013	0.016	.0168	.0045
LAB- 5 (COMB)	0.01	0.02	0.03	0.01		.0175	.0096
LAB- 6 (GRAV)	0.007	0.007	0.003	0.006	0.009	.0064	.0022
LAB- 7 (GRAV)	0.03	0.03	0.04	0.02	0.02	.0280	.0084
LAB- 9 (GRAV)	0.07	0.08	0.06	0.07	0.06	.0680	.0084
LAB-11 (GRAV)	0.082	0.086	0.082	0.086	0.082	.0836	.0022
LAB-15 (GRAV)	0.04	0.05	0.06	0.05	0.05	.0500	.0071
LAB-16 (TURBID)	0.013	0.012	0.014	0.014	0.013	.0132	.0008
*LAB-17 (COMB)	0.782	0.697	0.760	0.691	0.662	.7184	.0504
LAB-18 (GRAV)	0.03	0.035	0.03	0.03	0.03	.0310	.0022

**Outliers, not used for computations*

Table A-5a – Laboratory results, means and standard deviations for total iron and titanium in UTS-4

FE(TOTAL)

						MEAN	S.D.
LAB- 1 (PLASMA)	2.68	2.60	2.64	2.68	2.64	2.6480	.0335
LAB- 2 (AA)	2.69	2.72	2.69	2.69	2.69	2.6960	.0134
LAB- 3 (AA)	2.69	2.69	2.69	2.59	2.69	2.6700	.0447
LAB- 3 (PLASMA)	2.56	2.58	2.52	2.52	2.56	2.5480	.0268
LAB- 4 (AA)	2.56	2.56	2.56	2.60	2.56	2.5680	.0179
*LAB- 5 (AA)	2.90	3.00	3.00	2.95	2.95	2.9600	.0418
LAB- 6 (TITR)	2.53	2.54	2.55	2.52	2.53	2.5340	.0114
LAB- 6 (AA)	2.64	2.61	2.63	2.61	2.64	2.6260	.0152
LAB- 7 (AA)	2.66	2.51	2.75	2.73	2.72	2.6740	.0976
*LAB- 8 (AA)	3.16	3.14	3.13	3.24	3.16	3.1660	.0434
LAB- 9 (PLASMA)	2.50	2.46	2.54	2.25*	2.51	2.4520	.1165
LAB-10 (XRF)	2.755	2.765	2.755	2.73	2.73	2.7470	.0160
LAB-11 (PLASMA)	2.59	2.65	2.56	2.58	2.60	2.5960	.0336

TITANIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	0.26	0.26	0.27	0.28	0.27	.2680	.0084
LAB- 2 (AA)	0.18	0.22	0.22	0.22	0.18	.2040	.0219
LAB- 3 (AA)	0.22	0.22	0.22	0.22	0.22	.2200	.0000
LAB- 3 (PLASMA)	0.212	0.212	0.195	0.184	0.214	.2034	.0133
LAB- 4 (COLOR)	0.28	0.28	0.26	0.29	0.28	.2780	.0110
LAB- 5 (COLOR)	0.24	0.24	0.24	0.24	0.21	.2340	.0134
LAB- 6 (COLOR)	0.258	0.259	0.259	0.259	0.259	.2588	.0004
LAB- 7 (AA)	0.20	0.19	0.19	0.18	0.19	.1900	.0071
LAB- 8 (AA)	0.29	0.30	0.29	0.30	0.29	.2940	.0055
LAB- 9 (PLASMA)	0.26	0.26	0.26	0.26	0.27	.2620	.0045
LAB-10 (XRF)	0.26	0.265	0.265	0.26	0.26	.2620	.0027
LAB-11 (PLASMA)	0.196	0.186	0.184	0.186	0.189	.1882	.0047

Outliers, not used for computations

Table A-5b – Laboratory results, means and standard deviations for aluminum and calcium in UTS-4

ALUMINUM

						MEAN	S.D.
LAB- 1 (PLASMA)	6.09	5.98	6.03	6.03	5.93	6.0120	.0602
LAB- 2 (AA)	6.192	6.293	6.192	6.192	6.192	6.2122	.0452
*LAB- 3 (AA)	5.906	5.938	5.906	5.832	5.906	5.8976	.0392
LAB- 3 (PLASMA)	6.35	6.35	6.35	6.25	6.35	6.3300	.0447
LAB- 4 (AA)	6.20	6.20	6.20	6.28	6.24	6.2240	.0358
LAB- 5 (AA)	6.30	6.57	6.51	6.35	6.51	6.4480	.1163
LAB- 6 (AA)	6.30	6.36	6.37	6.33	6.33	6.3380	.0277
LAB- 7 (AA)	6.43	6.37	6.48	6.48	6.48	6.4480	.0487
LAB- 8 (AA)	6.48	6.44	6.41	6.49	6.46	6.4560	.0321
LAB- 9 (PLASMA)	6.31	6.26	6.28	6.32	6.34	6.3020	.0319
LAB-10 (XRF)	6.20	6.22	6.215	6.195	6.215	6.2090	.0108
LAB-11 (PLASMA)	6.18	6.21	6.12	6.18	6.25	6.1880	.0476

CALCIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	1.53	1.52	1.55	1.57	1.57	1.5480	.0228
*LAB- 2 (AA)	6.64	6.74	6.64	6.64	6.64	6.6600	.0447
LAB- 3 (AA)	1.80	1.80	1.80	1.80	1.80	1.8000	.0000
LAB- 3 (PLASMA)	1.72	1.79	1.75	1.75	1.77	1.7560	.0261
LAB- 4 (AA)	1.68	1.68	1.64	1.68	1.68	1.6720	.0179
LAB- 5 (AA)	1.75	1.71	1.71	1.75	1.71	1.7260	.0219
LAB- 6 (AA)	1.71	1.74	1.72	1.73	1.74	1.7280	.0130
LAB- 7 (AA)	1.89	1.89	1.90	1.90	1.89	1.8940	.0055
LAB- 8 (AA)	1.82	1.84	1.84	1.87	1.83	1.8400	.0187
LAB- 9 (PLASMA)	1.74	1.71	1.70	1.75	1.78	1.7360	.0321
LAB-10 (XRF)	1.81	1.805	1.805	1.80	1.80	1.8040	.0042
LAB-11 (PLASMA)	1.73	1.77	1.73	1.73	1.74	1.7400	.0173

**Outliers, not used for computations*

Table A-5c – Laboratory results, means and standard deviations for barium and uranium in UTS-4

BARIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	65.0	72.0	75.0	68.0	73.0	70.60	4.0373
LAB- 4 (XRF)	30.	20.	40.	30.	30.	30.00	7.0711
LAB- 5 (XRF)	70.0	90.0	70.0	100.0	90.0	84.00	13.4164
LAB- 6 (AE)	90.0	90.0	90.0	90.0	90.0	90.00	0.0000
LAB- 7 (AA)	50.	70.	50.	100.	60.	66.00	20.7364
LAB- 8 (AA)	16.0	17.0	15.0	15.0	15.0	15.60	.8944
LAB- 9 (PLASMA)	73.0	71.0	101.0	74.0	74.0	78.60	12.5817
LAB-10 (XRF)	80.	100.	90.	90.	90.	90.00	7.0711
LAB-11 (PLASMA)	80.3	80.8	90.6	80.8	81.7	82.84	4.3673
LAB-15 (XRF)	48.	49.	48.	48.	48.	48.20	.4472
LAB-17 (AA)	80.	77.	54.	34.	34.	55.80	22.2980
LAB-18 (XRF)	60.	70.	60.	80.	90.	72.00	13.0384

URANIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	1065.0	1053.0	1020.0	1095.0	1080.0	1062.60	28.5710
LAB- 2 (FLUOR)	900.0	950.0	1000.0	1050.0	950.0	970.00	57.0088
LAB- 2 (NAA)	989.0	991.0	989.0	991.0	994.0	990.80	2.0494
LAB- 3 (FLUOR)	1050.0	1025.0	1050.0	1050.0	1050.0	1045.00	11.1803
LAB- 3 (NAA)	991.0	996.0	985.0	998.0	999.0	993.80	5.8052
LAB- 4 (FLUOR)	983.	988.	982.	990.	989.	986.40	3.6469
LAB- 5 (FLUOR)	965.0	960.0	930.0	950.0	980.0	957.00	18.5742
LAB- 6 (NAA)	1094.0	1054.0	1129.0	1036.0	1051.0	1072.80	38.0487
LAB- 6 (NAA)	1079.	1076.	1057.	1059.	1043.	1062.80	14.8054
LAB- 7 (FLUOR)	930.	910.	1000.	950.	955.	949.00	33.6155
LAB- 8 (FLUOR)	1047.0	1046.0	1049.0	1049.0	1035.0	1045.20	5.8481
LAB- 9 (FLUOR)	1015.0	1015.0	1045.0	1125.0*	1025.0	1045.00	46.3681
LAB-10 (NAA)	1039.	1037.	1028.	1036.	1035.	1035.00	4.1833
LAB-11 (FLUOR)	930.	920.	1000.	955.	950.	951.00	30.9031

Outliers, not used for computations

Table A-5d – Laboratory results, means and standard deviations for thorium and total sulphur in UTS-4

THORIUM

						MEAN	S.D.
LAB- 1 (PLASMA)	21.0	21.0	23.0	22.0	22.0	21.80	.8367
LAB- 2 (NAA)	14.0	15.0	14.0	14.0	14.0	14.20	.4472
LAB- 3 (NAA)	15.0	14.0	14.0	14.0	14.0	14.20	.4472
* LAB- 3 (PLASMA)	38.2	38.1	31.2	31.2	38.2	35.38	3.8160
LAB- 4 (XRF)	14.	16.	14.	15.	12.	14.20	1.4832
LAB- 5 (XRF)	21.0	21.0	20.0	20.0	22.0	20.80	.8367
LAB- 6 (NAA)	9.0	18.0	18.0	18.0	18.0	16.20	4.0249
* LAB- 7 (COLOR)	62.	64.	60.	58.	61.	61.00	2.2361
LAB- 8 (COLOR)	13.3	9.4	13.0	14.7	14.1	12.90	2.0676
LAB-10 (NAA)	14.5	14.5	14.3	14.6	14.4	14.46	.1140
LAB-11 (NAA)	17.0	16.0	17.0	16.0	16.0	16.40	.5477
LAB-12 (NAA)	9.2	8.6	7.8	9.3	9.9	8.96	.7956
LAB-13 (NAA)	14.8	15.0	17.6*	15.2	14.7	15.46	1.2116
LAB-14 (NAA)	15.0	15.4	15.5	15.0	15.9	15.36	.3782

S(TOTAL)

						MEAN	S.D.
LAB- 1 (TITR)	1.840	1.740	1.750	1.810	1.770	1.7820	.0421
LAB- 2 (TITR)	1.7	1.7	1.7	1.7	1.7	1.7000	.0000
LAB- 3 (TITR)	1.7	1.7	1.7	1.7	1.7	1.7000	.0000
LAB- 4 (TITR)	1.86	1.91	1.91	1.86	1.86	1.8800	.0274
LAB- 5 (TITR)	1.72	1.72	1.78	1.76	1.74	1.7440	.0261
LAB- 6 (GRAV)	1.761	1.814	1.867	1.708	1.760	1.7820	.0605
LAB- 7 (GRAV)	1.84	1.84	1.88	1.84	1.82	1.8440	.0219
LAB- 8 (COLOR)	1.77	1.79	1.79	1.83	1.78	1.7920	.0228
LAB- 9 (TITR)	1.85	1.84	1.81	1.80	1.83	1.8260	.0207
*LAB-10 (XRF)	3.12	3.19	3.13	3.105	3.12	3.1330	.0331
*LAB-11 (TITR)	2.27	2.30	2.26	2.35	2.25	2.2860	.0404
LAB-15 (COMB)	1.76	1.77	1.75	1.73	1.72	1.7460	.0207
LAB-16 (GRAV)	1.88	1.86	1.84	1.87	1.88	1.8660	.0167
LAB-17 (COMB)	2.17	1.93	2.01	1.84	1.82	1.9540	.1426
LAB-18 (COMB)	1.794	1.795	1.794	1.797	1.792	1.7944	.0018

Outliers, not used for computations

Table A-5e – Laboratory results, means and standard deviations for sulphate and nickel in UTS-4

SULPHATE

						MEAN	S.D.
LAB- 1 (GRAV)	5.231	4.913	4.973	5.183	4.943	5.0486	.1471
LAB- 4 (TITR)	5.30	5.35	5.25	5.30	5.33	5.3060	.0378
*LAB- 5 (COMB)	1.72	1.75	1.74	1.72	1.78	1.7420	.0249
LAB- 6 (GRAV)	5.270	5.303	5.250	5.354	5.248	5.2850	.0445
LAB- 7 (GRAV)	5.26	5.25	5.27	5.25	5.28	5.2620	.0130
LAB- 8 (COLOR)	3.61	3.60	3.63	3.62	3.63	3.6180	.0130
LAB- 9 (GRAV)	5.17	5.10	5.12	5.15	5.10	5.1280	.0311
LAB-10 (GRAV)	5.28	5.20	5.40	5.20	5.275	5.2710	.0819
LAB-11 (GRAV)	5.39*	5.27	5.28	5.24	5.27	5.2900	.0579
*LAB-15 (GRAV)	3.57	3.69	3.52	3.53	3.51	3.5640	.0740
LAB-16 (GRAV)	5.17	5.18	5.16	5.17	5.19	5.1740	.0114
*LAB-17 (COMB)	0.172	0.172	0.129	0.150	0.150	.1546	.0180
LAB-18 (GRAV)	5.16	5.17	5.20	5.18	5.19	5.1800	.0158

NICKEL

						MEAN	S.D.
LAB- 1 (PLASMA)	175.0	178.0	175.0	176.0	185.0	177.80	4.2071
LAB- 2 (AA)	85.0	85.0	85.0	85.0	85.0	85.00	0.0000
LAB- 3 (AA)	85.0	85.0	85.0	85.0	85.0	85.00	0.0000
LAB- 3 (PLASMA)	121.0	121.0	122.0	121.0	122.0	121.40	.5477
LAB- 4 (AA)	140.	150.	140.	140.	140.	142.00	4.4721
LAB- 5 (AA)	150.0	170.0	150.0	150.0	180.0	160.00	14.1421
LAB- 6 (AA)	150.0	150.0	150.0	150.0	150.0	150.00	0.0000
LAB- 7 (AA)	160.0	160.0	160.0	160.0	160.0	160.00	0.0000
LAB- 8 (AA)	194.0	197.0	206.0	209.0	201.0	201.40	6.1887
LAB- 9 (AA)	214.0	200.0	204.0	195.0	201.0	202.80	7.0498
LAB-10 (AA)	130.	130.	130.	130.	130.	130.00	0.0000
LAB-11 (PLASMA)	195.	194.	192.	194.	192.	193.40	1.3416

Outliers, not used for computations

Table A-5f – Laboratory results, means and standard deviations for arsenic in UTS-4

ARSENIC

						MEAN	S.D.
LAB- 1 (PLASMA)	44.0	43.0	44.0	39.0	41.0	42.20	2.1679
LAB- 4 (TITR)	34.0	32.0	39.0	32.0	39.	35.20	3.5637
LAB- 2 (AA)	33.6	32.8	32.0	33.6	32.0	32.80	.8000
LAB- 3 (AA)	36.8	36.8	36.8	33.6	34.4	35.68	1.5595
LAB- 5 (COLOR)	38.0	35.0	37.0	38.0	36.0	36.80	1.3038
LAB- 6 (NAA)	40.0	40.0	40.0	40.0	40.0	40.00	0.0000
LAB- 7 (COLOR)	35.0	40.0	37.0	31.0	33.0	35.20	3.4928
LAB- 8 (COLOR)	40.0	41.0	41.0	40.0	41.0	40.60	.5477
LAB- 9 (AA)	40.0	36.0	40.0	38.0	38.0	38.40	1.6733
LAB-10 (NAA)	40.5	41.0	42.0	40.5	41.	41.00	.6124
*LAB-11 (AA)	52.0	53.0	52.0	53.0	52.0	52.40	.5477

Outliers, not used for computations

Table A-6a – Summary of analytical methods for total iron

Laboratory	Decomposition	Separation, reagents, procedure	Finish
1	$LiBO_2$ fusion		ICP-AE
2,3b,6b	HNO_3 + $HClO_4$ + HF		AA
3a,11	HCl + HNO_3 + $HClO_4$ + HF		ICP-AE
4,5	Na_2O_2 fusion		AA
6a	Na_2O_2-NaOH fusion	dichromate titration	Titrimetry
7,8	Na_2O_2 or Na_2O_2 + Na_2CO_3 fusion		
9	Li_2CO_3 + H_3BO_3 fusion		ICP-AE
10	$Li_2B_4O_7$ button fusion		Xrf

Table A-6b – Summary of analytical methods for titanium

Laboratory	Decomposition	Separation, reagents, procedure	Finish
1	$LiBO_2$ fusion		ICP-AE
2,3b	HNO_3 + $HClO_4$ + HF		AA
3a,11	HCl + HNO_3 + $HClO_4$ + HF		ICP-AE
4,5	Na_2O_2 fusion	Ti color developed with H_2O_2	Colorimetry
6	$K_2S_2O_7$ fusion	Ti color developed with H_2O_2	Colorimetry
7,8	Na_2O_2 or Na_2O_2 + Na_2CO_3 fusion		AA
9	Li_2CO_3 + H_3BO_3 fusion		ICP-AE
10	$LiBO_2$ button fusion		Xrf

Table A-6c – Summary of analytical methods for aluminum

Laboratory	Decomposition	Separation, reagents, procedure	Finish
1	$LiBO_2$ fusion		ICP-AE
2,3b,4,5,6	HNO_3 + $HClO_4$ + HF		AA
3a,11	HCl + HNO_3 + $HClO_4$ + HF		ICP-AE
7,8	Na_2O_2 or Na_2O_2 + Na_2CO_3 fusion		AA
9	Li_2CO_3 + H_3BO_3 fusion		ICP-AE
10	$LiBO_2$ button fusion		Xrf

Table A-6d – Summary of analytical methods for calcium

Laboratory	Decomposition	Separation, reagents, procedure	Finish
1	$LiBO_2$ fusion		ICP-AE
2,3b,4,5,6	HNO_3 + $HClO_4$ + HF		AA
3a,11	HCl + HNO_3 + $HClO_4$ + HF		ICP-AE
7	HCl + HNO_3		AA
8	Na_2O_2 + Na_2CO_3 fusion		AA
9	Li_2CO_3 + H_3BO_3 fusion		ICP-AE
10	$LiBO_2$ button fusion		Xrf

Table A-6e – Summary of analytical methods for barium

Laboratory	Decomposition	Separation, reagents, procedure	Finish
1	$LiBO_2$ fusion		ICP-AE
4,5,18		Energy dispersive spectrometry	Xrf
6	Na_2CO_3 fusion		Flame emission
7	$LiBO_2$ fusion		AA
8	$Na_2O_2 + Na_2CO_3$ fusion		AA
9	$Li_2CO_3 + H_3BO_3$ fusion		ICP-AE
10		8:2 sample: binder pelletization	Xrf
11	$HCl + HNO_3 + HClO_4 + HF$		ICP-AE
16	$HF + H_2SO_4$; residue fused with Na_2CO_3	$BaCO_3$ precipitated; dissolved with HCl and precipitated as $BaSO_4$; millipore filtration	Xrf
17	$HNO_3 + HF + HCl$	$BaSO_4$ precipitated; filtered and fused with $LiBO_2$; leach with HNO_3	AA

Table A-6f – Summary of analytical methods for uranium

Laboratory	Decomposition	Separation, reagents, procedure	Finish
1	$HNO_3 + HClO_4 + HF$		ICP-AE
2,3a,4,5,7,8			
9,11	$HNO_3 + HClO_4 + HF$	NaF-LiF fusion disc	Fluorimetry
2b,3b		Delayed neutron counting	NAA
6a,6b,10		Instrumental neutron activation	NAA

Table A-6g – Summary of analytical methods for thorium

Laboratory	Decomposition	Separation, reagents, procedure	Finish
1,3a	$HNO_3 + HClO_4 + HF$		ICP-AE
2,3b,12,13,14		Slowpoke reactor, measure 312 keV peak of ^{233}Pa	NAA
4,5		Wave-length dispersive spectrometry	Xrf
6,10,11		Instrumental neutron analysis	NAA
7	Na_2O_2 fusion	The color developed with Arsenazo III	Colorimetry
8	$Na_2O_2 + Na_2CO_3$ fusion	The color developed with thoron	Colorimetry
9		Direct measurement	α-spectrometry

Table A-6h – Summary of analytical methods for total sulphur

Laboratory	Decomposition	Separation, reagents, procedure	Finish
1,2,3,4,5 8,9,11,15, 17,18	Combustion (Leco furnace)	SO_2 dissolved in HC1 + K1; I_2 titrated with KIO_3	Combustion-titrimetry
6,16	HNO_3 + Br_2; Na_2O_2 fusion of insolubles	$BaSO_4$ precipitation	Gravimetry
7	Na_2CO_2 + KNO_3 fusion	$BaSO_4$ precipitation	Gravimetry
10		8:2 sample: binder pellet Cr oxidation	Xrf
16	HC1 + HNO_3 + Br_2	Fe reduced with $H_2NOH.HC1$ $BaSO_4$ precipitated	Gravimetry

Table A-6i – Summary of analytical methods for sulphate

Laboratory	Decomposition	Separation, reagents, procedure	Finish
1,9,10,11, 15,16	Boiled with 10% HC1 for 1 h	$BaSO_4$ precipitation	Gravimetry
2,3	Sulphide minerals decomposed with HI + $HgC1_2$	H_2S absorbed as CdS; oxidized with excess I_2; remaining I_2 titrated with $Na_2S_2O_3$; sulphate is total sulphur – sulphide sulphur	Combustion-titrimetry
4,5	Sulphide minerals decomposed with H_3PO_4 + Sn	Residual sulphur by combustion (Leco furnace)	Combustion-titrimetry
6	Hot water leach	$BaSO_4$ precipitation	Gravimetry
7	Sulphide minerals decomposed with HC1; residue fused with Na_2CO_3 + KNO_3	$BaSO_4$ precipitation	Gravimetry
15,17,18	Boiled with 10% HC1 for 1 h; sulphur in residue determined by Leco-furnace-iodiometry	Sulphate in total sulphur minus sulphur after HC1 leach	Combustion-titrimetry
16	Boiled with 10% HC1 for 1 h	Dissolved sulphate precipitated as $BaSO_4$	Gravimetry for UTS-1 to UTS-3 Turbidimetry for UTS-4

Table A-6j – Summary of analytical methods for nickel

Laboratory	Decomposition	Separation, reagents, procedure	Finish
1,3a,11	HNO_3 + $HC10_4$ + HF		ICP-AE
2,3b,4,5 6,7,9,10	HNO_3 + $HC10_4$ + HF		AA
8	Na_2O_2 + Na_2CO_3 fusion		AA

Table A-6k – Summary of analytical methods for arsenic

Laboratory	Decomposition	Separation, reagents, procedure	Finish
1	$HNO_3 + HClO_4 + HF$		ICP-AE
2,3	$K_2S_2O_7$ fusion	Arsine vapor evolution	AA
4,5,8	$HNO_3 + HClO_4 + HF$	Arsine vapor absorbed in silver dithiocarbamate-pyridine	Colorimetry
6,10		Instrumental neutron activation	NAA
7	$HNO_3 + HClO_4 + HF$	Molyboarsenate separation by extraction into chloroform-butanol and stripping into aqueous phase	Colorimetry
9	$HCl + HNO_3$	Arsine vapor evolution	AA
11	$HNO_3 + HClO_4$	Arsine vapor evolution	AA

Table A-7a – Consensus values and related statistical parameters for UTS-1

Constituent	No. of sets of results	Total No. of results	Consensus value	95% CL		σ_A
				Low	High	
Fe(total)	12	60	4.87%	4.81	4.94	0.05
Ti	10	50	0.54%	0.51	0.57	0.01
Al	12	60	6.24%	6.15	6.33	0.06
Ca	11	55	5.24%	5.17	5.31	0.04
S(total)	10	50	1.00%	0.94	1.05	0.03
Sulphate	9	45	2.64%	2.58	2.71	0.04
Ba	11	55	324 µg/g	288	360	13
U	12	60	49 µg/g	44	54	3
Th	11	55	138 µg/g	130	147	6

Table A-7b – Consensus values and related statistical parameters for UTS-2

Constituent	No. of sets of results	Total No. of results	Consensus value	95% CL		σ_A
				Low	High	
Fe(total)	12	60	3.20%	3.09	3.30	0.04
Ti	12	60	0.18%	0.16	0.21	0.01
Al	12	59	2.71%	2.65	2.76	0.04
Ca	12	60	0.42%	0.39	0.44	0.01
S(total)	11	55	3.23%	3.13	3.33	0.05
Sulphate	10	50	0.84%	0.76	0.92	0.01
Ba	11	55	464 µg/g	425	504	12
U	10	50	56 µg/g	55	57	2
Th	12	60	174 µg/g	162	187	6

Table A-7c – Consensus values and related statistical parameters for UTS-3

Constituent	No. of sets of results	Total No. of results	Consensus value	95% CL Low	95% CL High	σ_A
Fe(total)	12	60	3.25%	3.19	3.31	0.04
Ti	11	55	0.23%	0.21	0.24	0.007
Al	12	60	5.80%	5.66	5.94	0.04
Ca	10	50	4.03%	3.96	4.10	0.04
S(total)	10	50	0.23%	0.21	0.24	0.01
Sulphate	9	44	0.04%	0.01	0.06	0.005
Ba	11	55	212 µg/g	192	232	8
U	13	65	513 µg/g	497	529	9
Th	11	55	10.0 µg/g	8.6	11.4	1.4

Table A-7d – Consensus values and related statistical parameters for UTS-4

Constituent	No. of sets of results	Total No. of results	Consensus value	95% CL Low	95% CL High	σ_A
Fe(total)	11	54	2.62%	2.57	2.67	0.03
Ti	12	60	0.24%	0.22	0.26	0.008
Al	11	55	6.29%	6.20	6.38	0.05
Ca	11	55	1.75%	1.69	1.81	0.02
S(total)	13	65	1.80%	1.76	1.84	0.04
Sulphate	9	44	5.21%	5.15	5.28	0.04
Ba	12	60	65 µg/g	50	80	10
U	14	69	1010 µg/g	984	1036	19
Th	12	59	15.4 µg/g	13.2	17.5	1.0
Ni	12	60	151 µg/g	125	176	5
As	10	50	38 µg/g	36	40	2

Table A-8 – Approximate chemical composition values

Constituent	UTS-1	UTS-2	UTS-3	UTS-4
	mass %			
SiO_2	61.9	84.0	65.4	57.8
Na_2O	5.0	0.1	5.1	0.2
K_2O	2.0	2.0	0.3	0.4

PART B

RADIOISOTOPE ACTIVITIES IN UTS-1 to UTS-4

INTERLABORATORY PROGRAM FOR RADIOISOTOPES

A fifth material, designated UTS-5, was incorporated in the interlaboratory program. This material was produced by diluting CCRMP compositional reference ore (DH-1a) that contains certified amounts of uranium, thorium, and radium-226 (3,B1) with precipitated silica powder. Secular equilibrium in both the uranium and thorium decay series is probable. Consequently, UTS-5 was incorporated as a 'yardstick' of the probable quality of the interlaboratory program, and also to test the feasibility of silica dilution or possible future control applications. Participants were not informed of the nature of UTS-5.

Eight Canadian laboratories contributed isotope activity measurements, seven of these under contract arrangements with Supply and Services Canada (Table B-1). All participants were experienced with radiochemical determinations in uranium tailings.

It was requested that isotope activities (becquerels per gram) be measured in four independently-treated subsamples of each material. Recognized reference standards having published total uncertainties (including a precision estimate at the 99% confidence level) of less than 5% were to be employed in determining instrumental calibration factors for the measurements. Sufficient count data were to be accumulated that the uncertainty component in each result due to counting statistics was less than 2% (at 1σ confidence). The expertise of the participants was otherwise relied upon to provide unbiased and precise results within their capabilities.

The radioactive isotopes measured in this project were thorium-230, radium-226, lead-210, and polonium-210 (uranium-238 decay series); and thorium-232, radium-228, and thorium-228 (thorium-232 decay series, measured in UTS-1, UTS-2 and UTS-5 only). In addition, one laboratory provided measurements of thorium-232 and thorium-228 in UTS-3 and UTS-4, and protactinium-231 in all materials.

The analytical results are presented in Tables B-2 to B-11. The calibration standards are identified in Table B-14. Radiochemical methodologies are summarized in Table B-12; contractor reports and references cited should be consulted for more detailed descriptions.

CONSENSUS ACTIVITY VALUES: STATISTICAL TREATMENT OF RESULTS

The grand mean of all individual determinations exclusive of statistical outlier sets is defined as the consensus value for the activity of an isotope for purposes of this program. Dixon's 'r' statistic for the extreme laboratory mean values was used to identify laboratory sets having a low probability of belonging to the assumed consensus universe (B2). A level of significance $\alpha = 0.05$ ($r_{.05}$) was used for this purpose. The suspected sets were excluded from the calculation of the consensus value and its uncertainty. Four instances of such apparent outliers at this confidence level were detected in the 29 consensus determinations herein (Table B-17). However, the positive result in one case (Ra-228 in UTS-1) was deemed to arise from a fortuitously small variance in the small set of peer means, and was consequently not declared as an outlier set.

Results of analysis of variance (Tables B-15 and B-16) indicate that the between-laboratory component is generally significant relative to the within-laboratory component as has been observed to be the norm in chemical analysis consensus programs.

The consensus activity values for UTS-1 to UTS-4, their uncertainty estimates, and associated statistical parameters are compiled in Table B-18. The confidence limits (CL) estimate the range of reproducibility of the mean in 95% of cases were the identical program to be repeated many times. The average within-laboratory relative standard deviation ($\overline{CV}$, %) and the relative standard deviation of the individual determinations from the grand mean (RSD, %) provide an indication of how closely an individual determination approaches respectively, the laboratory estimate and consensus value on the average for general comparative purposes.

The corresponding results for UTS-5 are compiled separately in Table B-19; a 'bias estimate' comparing the consensus and predicted activity values is included for this material.

DISCUSSION

Results for UTS-5

UTS-5 was incorporated in the consensus program as a 'blind control' material to indicate potential biases in laboratory methods and consequently in the consensus values. Gamma-ray and uranium concentration measurements indicated that a homogeneous dilution of DH-1a (49.4 ± .6%, 2σ) in the silica was accomplished in the blending of this material (B3). From the certified uranium and thorium concentrations in DH-1a and published half-lives, the activities of uranium-238, uranium-235 and thorium-232 in UTS-5 are 15.86 ± .32, 0.744 ± .018, and 1.82 ± .10 Bq g^{-1} respectively, with uncertainty estimates propagated at the 99% confidence level (B1,B4,B5,B6). The consensus value for radium-226 in DH-1a in a thirteen-laboratory consensus program was 31.5 ± 1.1 Bq g^{-1} essentially confirming uranium-radium secular equilibrium (3). It is inferred that there is a high probability that thorium-230 is also in secular equilibrium. Radon emanation rate measurements of DH-1a suggest that lead-210 and polonium-210 may be 3 to 5% or more below secular equilibrium values if these rates existed in the natural ore state (B7,B8,B9). Polonium-210 in DH-1a was measured as 31.3 ± 1.1 Bq g^{-1} (95% CL), however, suggesting equilibrium (B9). In view of the relatively short half-lives of radium-228 and thorium-228 and the nature of DH-1a, it is highly probable that these isotopes are in secular equilibrium with thorium-232.

The consensus program results for UTS-5 are compared with the predicted (secular equilibrium) conditions in Table B-19. In all cases, the consensus means are in agreement with the predicted values, within the 95% confidence limits. The 'bias estimate', i.e., the relative departure of the consensus mean from the predicted value, is well within the 95% confidence interval estimate for each isotope. The bias estimate approaches the 95% confidence limit only for polonium-210. The single laboratory result for protactinium-231 suggests either a slightly low methodological bias or a lower than equilibrium activity for this isotope in UTS-5.

It is inferred that the consensus means, within the statistically determined confidence intervals, are likely to provide reliable estimates of the activities present in the tailing materials, provided that very different matrix or spectral interference effects are not encountered.

Results for UTS-1 to UTS-4

The recommended values for isotope activities for the tailings reference materials UTS-1 to UTS-4 are compiled in Table B-18.

The recommended activities of uranium-238 decay series isotopes in UTS-1 are not significantly different, i.e., their 95% confidence intervals overlap at about 3.5 Bq g^{-1}. Thus thorium-230, radium-226, lead-210, and polonium-210 are close to, if not in, secular equilibrium in UTS-1. Thorium may be slightly depleted relative to radium in UTS-2 ($^{230}Th/^{226}Ra$ = 0.8 ± 0.2), as may be lead-210 and polonium-210.

Thorium-230 is found to be depleted relative to radium-226 in UTS-3 and UTS-4 with activity ratios of .85 ± .07 and .59 ± .08 (2σ) respectively. Thorium-230 is similarly depleted relative to lead-210 in UTS-4 (Th/Pb .71 ± .14). Within the measurement uncertainties, polonium-210 is completely supported by lead-210 in both materials (activity ratios of .94 ± .09 and .95 ± .18 respectively). Similarly lead-210 may be completely supported by radium-226 in UTS-3 (activity ratio of .95 ± .06). Lead-210 may be slightly depleted in UTS-4 ($^{210}Pb/^{226}Ra$ = 0.84 ± 0.09).

There is no significant evidence for disequilibrium between thorium-232, radium-228, and thorium-228 in UTS-1 or UTS-2. It was decided to incorporate only the α-spectrometry measurements of thorium-232 in the radiochemistry consensus program, as neutron activation measurements of thorium are being incorporated in the chemical analysis certification program for these materials. Thorium-232 activities, calculated from chemical thorium values (Part A, Table 2) and the specific activity value 4.04 Bq mg^{-1} (B5), are 0.56 and 0.70 respectively for UTS-1 and UTS-2. These are about 25% lower than the recommended activities derived from radiochemical measurements; however the uncertainties allow for substantial agreement of both sets of results.

Comments on Precision and Accuracy

The confidence intervals generally indicate the current extent of agreement between participating laboratories as to the activities that are present in these Canadian tailings materials. On the average (over the five materials) the relative 95% confidence intervals are radium-226 (±6.5%), thorium-230, lead-210, and polonium-210 (all about ±13%), thorium-232 by alpha spectrometry (±19%), thorium-228 (±24%), and radium-228 (±34%)*.

The relative standard deviations of individual values from the consensus means provide a comparative indication of how closely a single determination can be expected to approach an interlaboratory consensus value at present (Tables B-18 and B-19). Averaged over the five materials, these are 17, 10, 10 and 12% respectively for thorium-230, radium-226, lead-210 and polonium-210. Averaged over the three materials the values are 19, 20 and 22% for thorium-232 (alpha-spectrometry), radium-228 and thorium-228.

**The confidence intervals are of course weighted by the numbers of determinations and laboratories which are considerably different for the uranium-238 series and thorium-232 series isotopes in this program (11,B2).*

Similarly, the values of average within-laboratory relative standard deviation ($\overline{CV}$, %) indicate how closely an individual determination may be expected to approach the mean value of a number of measurements within an 'average' participating laboratory. Averaged over the materials, the values are 10, 6, 7 and 5% respectively and 12, 11 and 10% respectively for the uranium-238 series and thorium-232 series as above.

With the exception of lead-210 determinations, no consistent evidence for relative methodological bias between laboratories could be deduced from an examination of laboratory mean values for all materials. In the case of lead-210 the mean results from laboratory 2 were consistently the highest in all materials. If relative bias exists in other determinations, it is sufficiently small to be masked by the within-laboratory mean uncertainties. Some partial trends are observed; however, these are inconclusive in view of the small amount of data. That the laboratory mean values are otherwise apparently randomly distributed lobbies in favour of the hypothesis that the consensus mean, within its statistical uncertainty, is the best available estimator of the true value from the program data. The validity of this approach is supported by the results for UTS-5.

Note must be taken, however, of the results found to lie outside of the consensus, particularly in view of the strong between-laboratory components of variance and the statistically small number of laboratories involved in the program. Inclusion of sets declared as outliers would result in recommended values remaining within the current 95% confidence intervals, but would approximately double these uncertainties. The apparent disagreements do not appear to stem from consistently observed methodological or calibration differences.

The relatively high degree of between-laboratory accord in the determinations of radium-226 is noteworthy. This probably arises from a combination of the use of established separation and tracer methodologies and a generally higher degree of experience with the isotope than with others, the use of common-source high-precision calibration standards, and possibly a less idiosyncratic nature of radium-226 towards matrix and spectral interference problems.

The accuracy of the results herein, of course, can be no better than the accuracy of the instrumental calibrations by which they were obtained. Calibrations, for the most part, were obtained using commercially available isotope reference materials bearing certified total uncertainties (Table B-19). The uncertainties in the calibration standards are not explicitly incorporated in the statistical uncertainties reported herein. Any significant differences in calibration parameters, laboratory-to-laboratory, would of course be reflected in the between-laboratories variance and hence be incorporated in the recommended value uncertainty. Such consistent biases are apparently masked by larger within-laboratory variances with the one exception noted above

The NBS radium standards used in the present work are certified as to radium mass; however, the uncertainty estimated for the specific activity and half-life of radium-226 of $\pm 0.44\%$ (3σ) increases the relative uncertainty in the calculated activity only slightly (B10).

Calibration solutions containing thorium-232, radium-228 and thorium-228 prepared from the 1906-refined thorium nitrate hydrated salt bear activity uncertainties propagated from uncertainties in the mass of thorium present* and the specific activity of thorium-232 (B11,B12). The activity uncertainties of all three isotopes are probably less than 5% in view of the age of the salt. This material (AM TYS-1) is employed as a calibration source for the alpha-spectrometry determinations of thorium isotopes and the beta-counting determination of radium-228 by several participants.

Laboratory 4 determined the activity of a thorium-228 solution relative to that of thorium-232 in a thorium nitrate solution from alpha activity ratios measured in a standard additions procedure. The thorium-230/thorium-228 activity ratios in spiked samples were then used to deduce thorium-230 activities. The ratios in unspiked samples were used to correct for the natural thorium-228 present, however, this interference was negligible except in the case of UTS-5. The activities of thorium-232 and thorium-228 in UTS-1, UTS-2 and UTS-5 were determined relative to thorium-230 from the alpha activity ratios from the unspiked samples.

Secular equilibrium assumptions are implicit in the use of chemical calibration standards by laboratory 1 (monazite sand for thorium-228 and radium-228), laboratory 4 (aged thorium oxide for radium-228), and laboratory 7 (BL-5 for thorium-230). The assumptions in the former two cases are probably reasonable from age considerations, and in the latter case by inference from indicated radium-226/uranium-238 secular equilibrium (3).

Laboratory 8 employed commercial americium-241 and lead-210 thin sources to determine the geometry/efficiency calibration for its alpha spectrometer and beta proportional counter respectively. Calibrations were confirmed with a standard radium-226 solution and other sources. This laboratory reported a problem with apparent changes in the activity concentration of radium-226 standard solution stored in a polyethylene bottle.

Calibrations for the determinations of lead-210 and polonium-210 were otherwise exclusively performed with commercial certified solutions

**One lot of this material was found to contain about 3.4% of sulphate and 40.75 ± .05% thorium vs. 39.45% for the formulation $Th(NO_3)_4\ 6H_20$ (B11).*

of lead-210/bismuth-210/polonium-210 (AM RBZ), polonium-210, or polonium-208. Polonium is notorious for plating out on containers and the RBZ reference solution certificate bears a caveat that there is no guarantee that the polonium remains in solution. If this occurred in calibrating solutions but not correspondingly in samples, a high bias in reported values would result. This is not indicated in the results for UTS-5.

No obvious general correlation was noted between the magnitude of activity (or nature of the material) and the magnitude of uncertainty of a given isotope. Laboratory 5 indicated that within-laboratory reproducibilities experienced for thorium-230 and radium-226 in UTS-4 were poorer than expected, and that gamma-ray measurements of radium-226 in UTS-3 and UTS-4 differed significantly from alpha spectrometry results, both for unknown reasons. Similarly, laboratory 6 reported 'poorer-than-expected' precisions or extended replicate determinations of radium-226 and lead-210 in UTS-2. Laboratory 8 found that natural barium in some materials degraded their alpha spectral resolution, sufficiently that tailing corrects were required, thereby raising the uncertainty.

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Table B-1 – Contributing laboratories for radiochemical determinations*

Lab-1**	Nuclear Activation Services Hamilton, Ontario (E.L. Hoffman)
Lab-2**	Chemex Laboratories Ltd. North Vancouver, British Columbia (M. Legeyt)
Lab-3	Monenco Analytical Laboratories Calgary, Alberta (J. Dean, N. Chiu) Contract 15SQ.23440-2-9145-4
Lab-4	Physics Department, University of Calgary Calgary, Alberta (C.J. Bland, P. Jarvis) Contract 15SQ.23440-2-9145-5
Lab-5	Saskatchewan Research Council Saskatoon, Saskatchewan (G. Smithson, V. Penner, M. Knelson, R. Ortlepp, L. MacDonald) Contract 15SQ.23440-2-9145-1
Lab-6	Eldorado Resources Ltd., Research and Development Division Ottawa, Ontario (M.C. Miedema, G.A. Dunlop, R. Jones, G.B. Moodie) Contract 15SQ.23440-2-9145-2
Lab-7	CANMET, Mineral Sciences Laboratories, Chemical Laboratory Ottawa, Ontario (J.L. Dalton, R. McCorkell, M. DeGagne)
Lab-8	Waterloo Research Institute. University of Waterloo Waterloo, Ontario (H. Sharma, B. Hauk) Contract 15SQ.23440-2-9145-6

Supply and Services Canada contracts as indicated, contractor reports filed with CANMET Research Program Office and National Uranium Tailings Research Program Office.

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Table B-2 – Analytical results, laboratory means and standard deviations for ^{238}U-series isotopes in UTS-1

	Bq g^{-1}						Mean	S.D.
^{230}Th Lab-2 (α)	2.37	2.74	2.59	2.48			2.545	.158
Lab-3 (α)	3.52	4.81	3.52	5.37			4.305	.935
Lab-4 (α)	4.91	4.56	3.70	4.37			4.385	.509
Lab-5 (α)	3.83	3.46	4.04	3.64			3.743	.249
Lab-6 (α)	3.01	3.15	3.21	3.02			3.098	.098
Lab-7 (α)	2.59	3.77	4.11	3.29			3.440	.659
Lab-8 (α)	3.50	3.63	3.77	3.79			3.673	.135
^{226}Ra Lab-1 (α)	2.9	3.3	4.8	3.7			3.68	.82
Lab-3 (α)	4.03	3.81	3.42	3.59			3.713	.265
Lab-4 (α)	3.88	3.60	3.80	4.13			3.853	.219
Lab-5 (gα)	3.53	3.53	3.47	3.48	3.49		3.500	.028
Lab-5 (γ)	3.49	3.52					3.505	.021
Lab-6 (Rn)	3.38	3.65	3.44	3.33			3.450	.141
Lab-7 (α)	3.81	3.40	3.96	3.85			3.755	.245
Lab-8 (α)	3.79	4.04	3.59	4.15			3.893	.252
^{210}Pb Lab-2 (β)	3.52	3.44	3.48	3.52			3.490	.038
Lab-3 (β)	3.00	3.03	3.18	3.00			3.052	.086
Lab-4 (α,^{210}Po)	3.33						3.33	
Lab-5 (β)	3.31	2.94	3.07	3.15	3.12	2.90	3.082	.149
Lab-6 (β)	3.27	3.24	3.23	3.15			3.223	.051
Lab-7 (β)*	2.52	2.40	2.52	1.81			2.313	.340
Lab-8 (β)	3.70	2.72	4.08	3.34			3.460	.579
^{210}Po Lab-2 (α)	3.44	3.22	3.26	3.03			3.238	.168
Lab-3 (α)	2.8	2.9	2.8	2.8			2.82	.05
Lab-4 (α)	3.60	3.77	3.46	3.34			3.542	.185
Lab-5 (α)	2.92	2.92	2.89	2.81	2.89	2.96		
	2.81						2.886	.057
Lab-6 (α)	3.00	2.81	3.03	2.85			2.922	.109
Lab-8 (α)	3.48	3.44	3.67	3.66			3.562	.120

Outlier set

Table B-3 – Analytical results, laboratory means and standard deviations for ^{238}U-series isotopes in UTS-2

	Bq g^{-1}						Mean	S.D.
^{230}Th Lab-2 (α)	3.26	3.22	3.44	3.33			3.312	.096
Lab-3 (α)	5.84	3.57	5.96	4.70			5.108	1.120
Lab-4 (α)	4.96	4.06	4.80	4.62			4.610	.392
Lab-5 (α)	3.87	4.08	3.78	3.80	3.72		3.850	.139
Lab-6 (α)	3.06	3.33	3.18	3.14			3.179	.113
Lab-7 (α)	4.74	4.66	4.37	4.63			4.600	.160
Lab-8 (α)	6.22	6.66	6.77	6.70			6.588	.249
^{226}Ra Lab-1 (α)	5.9	5.5	5.9	7.3			6.15	.79
Lab-3 (α)	6.36	4.91	4.74	4.55			5.140	.827
Lab-4 (α)	6.25	5.84	6.06	6.07			6.055	.168
Lab-5 (gα)	5.80	5.80	5.89	5.65			5.785	.100
Lab-5 (γ)	5.64	5.00					5.320	.453
Lab-6 (Rn)	5.01	6.06	5.06	7.01	4.77	6.19		
	5.68	3.48	5.76	5.13	5.42		5.416	.910
Lab-7 (α)	6.10	5.66	6.07	6.03			5.965	.205
Lab-8 (α)	5.35	4.72	5.06	4.88			5.003	.270
^{210}Pb Lab-2 (β)	4.85	4.70	4.60	5.33			4.870	.323
Lab-3 (β)	4.40	4.44	4.22	4.29			4.338	.101
Lab-4 (α,^{210}Po)	4.51						4.51	
Lab-5 (β)	4.62	4.67	4.49	4.65	4.93	4.71		
	4.69	4.82	4.85	4.76			4.721	.127
Lab-6 (β)	5.48	4.19	3.69	3.99	5.17	4.00		
	5.59	4.84	4.66	3.15	3.97		4.430	.778
Lab-7 (β)*	3.77	3.26	2.81	3.96			3.450	.519
Lab-8 (β)	3.82	3.75	4.75	5.24			4.390	.727
^{210}Po Lab-2 (α)	4.92	4.81	5.18	4.14			4.762	.443
Lab-3 (α)	4.2	4.3	3.66	4.5			4.165	.359
Lab-4 (α)	5.44	5.43	5.69	5.52			5.520	.120
Lab-5 (α)	3.88	4.25	3.88	3.88	4.37	4.07		
	4.44	4.37	4.00				4.127	.224
Lab-6 (α)	4.26	4.33	4.66	4.36			4.403	.177
Lab-8 (α)	4.11	3.85	3.48	3.50			3.735	.302

Outlier set

Table B-4 – Analytical results, laboratory means and standard deviations for ^{238}U-series isotopes in UTS-3

	Bq g^{-1}						Mean	S.D.
^{230}Th Lab-2 (α)	10.2	10.0	10.5	11.0			10.42	096
Lab-3 (α)	11.4	12.1	11.9	12.2			11.90	356
Lab-4 (α)	11.6	13.0	13.7	13.2			12.88	900
Lab-5 (α)	11.5	11.7	12.6	11.3	11.1		11.64	581
Lab-6 (α)	10.9	10.6	10.4	10.4			10.58	236
Lab-7 (α)	12.5	10.7	11.1	9.7			11.00	1.160
Lab-8 (α)	11.0	11.3	10.2	10.4			10.73	.512
^{226}Ra Lab-1 (α)	14.6	14.6	14.6	7.3			12.8	3.7
Lab-3 (α)	13.0	14.0	13.4	14.3			13.68	585
Lab-4 (α)	14.4	14.9	14.5	13.7			14.38	499
Lab-5 (gα)	12.9	12.5	12.8	12.9			12.78	189
Lab-5 (γ)	14.6	14.4					14.5	14
Lab-6 (Rn)	13.1	12.0	12.8	12.9			12.70	483
Lab-7 (α)	13.9	13.2	13.8	13.6			13.63	310
Lab-8 (α)	12.2	12.0	12.9	13.4			12.60	668
^{210}Pb Lab-2 (β)	13.1	13.9	14.1	13.3			13.60	476
Lab-3 (β)	11.5	12.1	12.3	13.2			12.28	704
Lab-4 (α,^{210}Po)	11.4						11.4	
Lab-5 (β)	12.8	12.4	11.5	12.3	13.3	12.2		
	13.4	13.7	14.0				12.84	814
Lab-6 (β)	12.0	12.7	11.9	12.7			12.32	435
Lab-7 (β)	10.8	12.8	14.8	9.29			11.92	2.397
Lab-8 (β)	13.9	13.2	13.1	11.9			13.02	830
^{210}Po Lab-2 (α)	13.2	13.6	13.7	12.7			13.30	455
Lab-3 (α)	11.7	10.1	11.8	10.9			11.12	793
Lab-4 (α)	11.8	11.0	11.2	11.2			11.30	346
Lab-5 (α)	11.84	11.84	11.84	11.65	12.02	11.84		
	12.21	12.21	11.47				11.88	241
Lab-6 (α)	11.11	9.99	11.03	10.99			10.78	529
Lab-8 (α)	12.8	13.1	12.3	12.3			12.62	395

Table B-5 – Analytical results, laboratory means and standard deviations for ^{238}U-series isotopes in UTS-4

	Bq g^{-1}						Mean	S.D.
^{230}Th Lab-2 (α)	22.5	23.4	22.8	23.2			22.98	.404
Lab-3 (α)*	40.8	42.7	36.6	41.5			40.40	2.652
Lab-4 (α)	24.2	24.2	24.5	23.1			24.00	.616
Lab-5 (α)	16.6	16.5	18.6	20.6			18.08	1.941
Lab-6 (α)	24.4	24.6	22.6	23.0			23.65	.998
Lab-7 (α)	27.2	17.9	27.1	24.6			24.20	4.369
Lab-8 (α)	23.6	26.2	24.1	24.7			24.65	1.127
^{226}Ra Lab-1 (α)	36.6	47.6	47.6	40.2			43.0	5.51
Lab-3 (α)	37.1	40.0	38.7	38.9			38.68	1.196
Lab-4 (α)	39.4	39.9	40.4	39.1			39.70	.572
Lab-5 (gα)	39.2	31.2	38.0	31.4	37.1	37.0		
	32.1						35.14	3.433
Lab-5 (γ)	35.9	35.6					35.75	.212
Lab-6 (Rn)	39.4	36.5	35.0	39.5			37.60	2.223
Lab-7 (α)	38.0	39.7	39.3	39.6			39.15	.785
Lab-8 (α)	40.5	39.4	42.7	40.7			40.82	1.374
^{210}Pb Lab-2 (β)	36.7	35.3	36.6	37.0			36.40	.753
Lab-3 (β)	33.0	30.7	32.0	33.7			32.35	1.303
Lab-4 (α, ^{210}Po)	30.6						30.6	
Lab-5 (β)	30.4	29.2	28.8	34.6	33.4	28.9		
	31.5						30.97	2.303
Lab-6 (β)	35.9	35.2	35.9	35.6			35.65	.332
Lab-7 (β)	30.0	31.4	35.8	32.6			32.45	2.473
Lab-8 (β)	28.8	28.2	26.9	29.7			28.40	1.175
^{210}Po Lab-2 (α)	34.8	34.6	34.5	38.0			35.48	1.688
Lab-3 (α)	23.5	25.4	23.0	24.5			24.10	1.068
Lab-4 (α)	35.3	37.8	32.1	37.4			35.65	2.608
Lab-5 (α)	29.6	30.3	28.9	28.1	31.1	27.8		
	29.6	29.6					29.38	1.090
Lab-6 (α)	33.6	34.0	33.9	36.4			34.48	1.295
Lab-8 (α)	27.9	27.4	26.9	27.6			27.45	.420

Outlier set

Table B-6 – Analytical results, laboratory means and standard deviations for ^{238}U-series isotopes in UTS-5

	Bq g^{-1}						Mean	S.D.
^{230}Th Lab-2 (α)	15.9	16.5	16.4	16.4			16.30	.271
Lab-3 (α)	14.6	14.8	14.8	14.1			14.52	.613
Lab-4 (α)	15.6	15.3	16.0	16.3			15.80	.440
Lab-5 (α)	15.7	15.6	15.9	15.8	15.3	15.8	15.60	.294
	15.1							
Lab-6 (α)	15.3	15.7	14.3	15.8			15.28	.685
Lab-7 (α)	16.1	18.5	16.6	18.2			17.35	1.179
Lab-8 (α)	13.0	12.4	13.4	12.2			12.75	.551
^{226}Ra Lab-1 (α)	21.6	22.0	18.3	18.3			20.05	2.027
Lab-3 (α)	13.9	14.2	14.7	15.3			14.52	.613
Lab-4 (α)	16.7	16.9	16.5	16.6			16.68	.171
Lab-5 ($g\alpha$)	15.3	16.3	16.7	16.6	15.1	15.0	15.83	.784
Lab-5 (γ)	17.8	17.5					17.65	.212
Lab-6 (Rn)	16.2	14.8	16.1	15.0			15.52	.727
Lab-7 (α)	15.6	16.3	15.8	16.5			16.05	.420
Lab-8 (α)	16.1	15.3	15.1	15.7			15.56	.444
^{210}Pb Lab-2 (β)	16.4	17.0	17.3	16.9			16.90	.374
Lab-3 (β)	13.4	13.9	13.8	14.8			13.98	.591
Lab-4 (α,^{210}Po)	13.8						13.8	
Lab-5 (β)	14.0	14.1	14.4	14.5	14.6	14.5		
	13.7						14.27	.327
Lab-6 (β)	14.3	14.8	14.7	14.7			14.62	.222
Lab-7 (β)	17.7	13.7	13.0	16.8			15.30	2.299
Lab-8 (β)	11.4	10.5	9.2	11.9			10.75	1.185
^{210}Po Lab-2 (α)	15.7	17.7	16.6	17.7			16.92	.967
Lab-3 (α)	11.3	10.4	11.5	14.2			11.85	1.638
Lab-4 (α)	14.2	14.8	15.2	14.5			14.68	.427
Lab-5 (α)	11.5	12.6	13.0	12.6	11.8	11.8		
	13.0						12.33	.618
Lab-6 (α)	14.5	15.1	16.2	15.5			15.32	.714
Lab-8 (α)	11.9	12.4	12.6	10.1			11.75	1.139

Table B-7 – Analytical results, laboratory means and standard deviations for ^{232}Th-series isotopes in UTS-1

	Bq g^{-1}						Mean	S.D.
^{232}Th Lab-1 (α)	.514	.579	.551	.555			.550	.027
Lab-2 (α)	.510	.586					.548	.054
Lab-2 (γ, daught)	.570	.570					.570	
Lab-3 (α)	.58	.82	.61	.93			.735	.168
Lab-4 (α)	.810	.774	.614	.717			.729	.086
Lab-5 (α)	.66	.55	.65	.65			.628	.052
Lab-5 (NAA)	.61						.61	
Lab-8 (α)	.75	.70	.73	.68			.715	.031
^{228}Ra Lab-1 (β)	<1.	<1.	<1.	<1.				
Lab-3 (β)	.74	.92	.74	.63			.758	.120
Lab-4 (γ)	.736						.736	
Lab-5 (β)	.54	.53	.55	.52	.57		.542	.019
Lab-8 (β)	.88	.71	.69	.75			.758	.085
^{228}Th Lab-2 (α)	.6	.6	.6	.6			.6	
Lab-3 (α)	.64	.67	.67	.92			.725	.131
Lab-4 (α)	.85	.79	.62	.75			.752	.097
Lab-5 (α)	.65	.58	.65	.65			.632	.035
Lab-8 (α)	1.22	.78	.77	.66			.858	.248

Table B-8 – Analytical results, laboratory means and standard deviations for ^{232}Th-series isotopes in UTS-2

	Bq g^{-1}						Mean	S.D.
^{232}Th Lab-1 (NAA)	.693	.656	.680	.680			.677	.015
Lab-2 (α)	.76	.69					.725	.050
Lab-2 (γ, daught)	.688	.692					.690	.003
Lab-3 (α)	.57	.65	1.20	.83			.812	.280
Lab-4 (α)	1.10	.85	.98	.98			.978	.102
Lab-5 (α)	.56	.68	.85	.84	.77		.740	.121
Lab-5 (NAA)	.74						.74	
Lab-8 (α)	.91	.91	1.27	1.26			1.088	.205
^{228}Ra Lab-1 (β)	<1.	<1.	<1.	<1.			<1.	
Lab-3 (β)	1.07	1.11	1.26	1.15			1.148	.082
Lab-4 (γ)	1.26						1.26	
Lab-5 (β)	.91	.80	.89	.80	.80	.85	.842	.050
Lab-8 (β)	1.18	1.16	1.23	1.09			1.165	.058
^{228}Th Lab-1 (α)	.6	.6	.6	.6			.6	
Lab-3 (α)	.85	.81	1.27	1.18			1.028	.232
Lab-4 (α)	1.07	.86	1.03	1.00			.990	.091
Lab-5 (α)	.56	.68	.87	.79	.74		.728	.117
Lab-8 (α)	1.23	1.29	1.21	1.44			1.293	.104

Table B-9 – Analytical results, laboratory means and standard deviations for ^{232}Th-series isotopes in UTS-5

	Bq g^{-1}						Mean	S.D.
^{232}Th Lab-1 (NAA)	1.89	1.89	1.88	1.86			1.880	.014
Lab-2 (α)	1.97	1.90					1.935	.050
Lab-2 (γ, daught)	1.94	1.88					1.910	.042
Lab-3 (α)	2.07	2.40	1.92	1.85			2.060	.244
Lab-4 (α)	2.42	2.31	2.34	2.43			2.375	.059
Lab-5 (α)	1.69	1.72	1.93	1.71			1.762	.112
Lab-5 (NAA)	1.76						1.76	
Lab-8 (α)	1.82	1.46	2.01	1.35			1.660	.308
^{228}Ra Lab-1 (β)	1.0	2.0	1.0	1.0			1.25	.500
Lab-3 (β)	2.2	2.2	2.1	2.6			2.28	.222
Lab-4 (γ)	2.95						2.95	
Lab-5 (β)	1.70	1.59	1.66	1.59	1.66	1.88		
	1.59						1.667	.104
Lab-8 (β)	1.95	1.73	1.80	1.85			1.833	.092
^{228}Th Lab-1 (α)	1.8	1.8	1.8	1.8			1.80	
Lab-3 (α)	1.97	2.40	2.04	1.85			2.065	.237
Lab-4 (α)	2.55	2.40	2.47	2.55			2.492	.072
Lab-5 (α)	1.69	1.72	2.02	1.98			1.852	.171
Lab-8 (α)	1.61	1.53	1.84	1.87			1.712	.168

Table B-10 – Analytical results and laboratory mean values for ^{232}Th and ^{228}Th in UTS-3 and UTS-4 (Lab-3 only, alpha spectroscopy)

		Bq g^{-1}				Mean	S.D.
UTS-3	^{232}Th	.15	.16	.22	.10	.16	.05
	^{228}Th	.13	.16	.23	.13	.16	.04
UTS-4	^{232}Th	.40	.56	.73	.48	.48	.07
	^{228}Th	.22	.27	.19	.26	.23	.04

Table B-11 – Analytical results and laboratory mean values for ^{231}Pa (Lab-3 only, alpha spectroscopy)

	Bq g^{-1}				Mean	S.D.
UTS-1	.23	.23	.20	.18	.21	.03
UTS-2	.30	.37	.51	.31	.37	.11
UTS-3	.54	.78	.61	.85	.70	.15
UTS-4	2.2	2.3	2.1	2.8	2.4	.2

Table B-12 – Summary of radiochemical procedures (a, b)

Lab-1 Decompositions of repeated multiple acid digestion; HC1, HNO_3, HF, H_2SO_4. Residues fused with $LiBO_2$. Dissolved in 10% HNO_3/10% HC1.

Separations: (volumetric aliquots)

^{226}Ra	– (c), α-spectroscopy.
^{228}Ra	– (c), (no ^{133}Ba), β-counting.
^{232}Th, ^{228}Th	– two-stage IX; SX (TTA), extract concentrated and evaporated on a steel planchet; α-spectroscopy; ^{232}Th also by NAA.

Lab-2 Decomposition by repeated multiple acid digestion; HNO_3, HF, $HC10_4$ (0.25 to 0.5 g).

Separations:

^{210}Pb	– (d) (1).
^{210}Po	– Ag deposition with ascrobic acid reductant (1), α-spectrometry.
^{230}Th	– Successive coprecipitations with lanthanum as hydroxide and fluoride, with Ba 'hold-back' carrier for Ra, final ppt. collected on 0.45 μm filter for α-spectrometry. ^{234}Th tracer by β-counting. ^{230}Th additions implied negligible self-absorption.

Lab-3 Decompositions: for Ra, Th: 2 g by KF and pyrosulphate fusion (2).
for Po, Pb: 1 g by repeated multiple digestions: HNO_3, HF, $HC10_4$.

Separations:

^{226}Ra	– (c), α-spectrometry.
^{228}Ra	– (e) (1, 3, 4, 5), ^{228}Ac β-counting.
^{230}Th, ^{232}Th, ^{228}Th	– supernate from (c) volumetrically diluted, and split into two equal portions, one spiked with ^{228}Th. Following SC (HDEHP) (5), thorium coprecipitated with cerous hydroxide, collected on 0.1 μm filter for α-spectrometry (6).
^{210}Pb	– (d) (1).
^{210}Po	– coprecipitation with PbS, dissolved in HC1, Ni deposition for α-spectrometry (7). ^{208}Po tracer used.

Lab-4 Decompositions by HF treatment followed by KF and pyrosulphate fusions of 1 g samples (8, 9).

Separations:

^{226}Ra	– (c), α-spectrometry.
^{232}Th, ^{228}Th, ^{230}Th	– coprecipitated with cerous hydroxide in presence of EDTA from supernate from (C) filtered onto 0.1 μm filter for α-spectrometry.
^{210}Po	– coprecipitated with tellerium in presence SO_2, $TiCl_3$, collected on 0.1 μm filter. (^{208}Po tracer used, α-spectrometry.)
^{228}Ra	– no separations, γ-spectrometry of ^{228}Ac on 40-75 g samples.
^{210}Pb	– indirectly, as supported ^{210}Po.

Table B-12 (Cont'd)

Lab-5 Decompositions of 1 g samples by repeated digestion with HF, H_2HSO_4 followed by pyrosulphate fusions. Radium samples dissolved in HNO_3, others in HC1.

Separations:

^{226}Ra	– (c) (1) (gross-alpha counting, with decay/ingrowth corrections), (also, gamma-spectrometry, 10 g).
^{228}Ra	– (e) (1, 5) (β-counting of ^{228}Ac).
^{210}Pb	– (d) (1) (β-counting of ^{210}Bi).
^{210}Po	– Ag deposition in presence of ascorbic acid and thioacetamide. (^{208}Po tracer used.)
^{232}Th, ^{228}Th, ^{230}Th	– a series of iron hydroxide and barium sulphate coprecipitation steps followed by SX (TTA), back extraction (2 M HNO_3), coprecipitation with cerous hydroxide with collection on a 0.1 μm filter of α-spectrometry. (^{234}Th tracer). (^{232}Th also by NAA).

Lab-6 Decompositions. for Ra by fusion with Rushing flux, $Na_2Co_3/K_2CO_2/Na_2B_4O_7$ (10). For Th, Pb by KF and pyrosulphate fusions (11). For Po by multiple acid digestion; HF, HNO_3, H_2SO_4, HC1 (1-2 g samples.)

Separations:

^{226}Ra	– ^{222}Rn emanated after 6 d. Storage of de-emanated solution in a sealed bubbler, ^{222}Rn and daughters measured by α-scintillation.
^{210}Pb	– (d).
^{210}Po	– Ni deposition from HC1 medium (^{208}Po tracer, α-spectrometry).
^{230}Th	– thorium in supernate from (c) coprecipitated with titanium hydroxide at pH 14 and filtered for alpha spectrometry.

Lab-7 Decompositions: for Ra, Th – repeated mixed acid digestion with HNO_2, HF, H_2SO_4, followed by pyrosulphate fusion and dissolution in HC1. For Pb – acid digestion with HNO_3, $HC1O_4$; residue treated with HF, H_2SO_4; dissolved in HC1. (All 1 g samples).

Separations:

^{226}Ra	– (c) (1), α-spectrometry.
^{210}Pb	– (d) (1), β-counting of ^{210}Bi, no recovery tracer.
^{230}Th	– coprecipitation with lanthanum as hydroxide and then fluoride followed by SX (TTA), back extraction (HNO_3) (1), coprecipitation with titanium hydroxide and filtration for alpha spectrometry.

Table B-12 (Cont'd)

Lab-8	Decompositions of 2 g samples by repeated acid digestion (HF, H_2SO_4) followed by pyrosulphate fusion and dissolution in 10% HC1.	
	Separations:	
	^{226}Ra	– (c).
	^{230}Th, ^{232}Th, ^{228}Th	– IX (cation, loaded pH 2-3, cations eluted with 12 M HC1, Th then eluted with 0.5 M oxalic acid) (13). Oxalate destroyed with HNO_3, SX (TTA), back-extracted into 2 M HNO_3 and Th isotopes electro-deposited (14) for α-spectrometry. (^{234}Th tracer used.)
	^{210}Pb	– IX eluent (above) passed through anion IX (to remove U) and Bi extracted by SX (DDTC), evaporated on planchet for β-counting 20 min and 1 d after extraction.
	^{228}Ra	– (e).
	^{210}Po	– Ag deposition, pH 0.3, 95°C, α-spectrometry, no tracer.

(a) Reports of contractors entitled 'Radiochemical determinations for tailings reference materials and references cited should be consulted for details (Table 1, Table 13).

(b) Abbreviations: IX, ion-exchange chromatography; SX, solvent extraction; TTA, theonyltrifluoroacetone; NAA, neutron activation analysis; HDEHP, bis(2-ethly-hexyl) phosphoric acid; DDTC, diethyldithiocarbanate; EDTA, ethlyenediamine-tetraacetic acid; DTPA, diethylenetriaminepentaacetic acid; AAS, atomic absorption spectrophotometry.

(c) Radium (and other radioisotope) are coprecipitated with lead sulphate; the precipitate is dissolved alkaline EDTA (or DTPA) and radium is selectively coprecipitated with barium sulphate which is collected on a membrane filter for α-spectrometry or gross alpha counting. Barium-133, added at the decomposition stage, is usually as a recovery tracer, employing gamma-ray spectrometry [e.g. (1)].

(d) Lead and bismuth carriers are added prior to decomposition; the dissolved sample is in 2M HC1. Bismuth SX (0.1% DDTC/$CHC1_3$); extracts evaporated, decomposed with HNO_3, and bismuth hydroxides precipitated (pH 8). The precipitate is dissolved in HC1, and BiOC1 (precipitated by dilution) is filtered for β-counting of ^{210}Bi after decay of other β-emitters. Correction is applied for decay of ^{210}Bi, and recovery is traced by AAS measurements of Bi carrier (1b).

(e) The Ba(Ra) SO_4 from (a) is redissolved in alkaline DTPA following a 3 d ingrowth of ^{228}Ac. Ba(Ra)SO_4 is removed by precipitation and ^{228}Ac is separated by SX (HDEHP/alkanes), back-extraction into 1M HNO_3 and coprecipitation with lanthanum oxalate which is filtered for β-counting (1, 5).

Table B-13 - References cited for radiochemical procedures

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1(b) Smithson, G., Fahri, M. and Petrow, M. "Radiochemical determination of lead-210 in environmental samples and samples resulting from uranium mining-milling operations"; ibid, Appendix B.

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12. Donaldson, E.M. "Methods for the analysis of ores, rocks and related materials"; Monograph 881, second edition; Ottawa, Supply and Services Canada; 1982.

13. Veska, E. "Origin and sub-surface migration of radionuclides from waste rock at an abandoned uranium mine near Bancroft, Ontario"; Ph.D. thesis, University of Waterloo (Canada); 1983.

14. Puphal, K.W. and Olsen, D.R. "Electrodeposition of alpha-emitting nuclides from a mixed oxalate-chloride electrolyte"; Anal Chem 44:284-289; 1972.

Table B-14 – Calibration reference materials and recovery tracers used for radiochemical measurements

	SEM (uncertainty, %)* [Tracer]						
	^{226}Ra	^{210}Pb	^{210}Po	^{230}Th	^{232}Th	^{228}Th	^{228}Ra
Lab 1	NBS 4964-B (0.5) [^{133}Ba]	–	–	–	– – – – – – – – – – st. monazite (1.2) – – – – – – – – – – [thorium] – – – – – – – – – – [cf. ^{226}Ra] – –		
Lab 2	–	AM RBZ (2.3) [Bi]	AM RBZ (2.3) [st. addn ^{210}Po]	IPL (6.) [^{234}Th]	–	–	–
Lab 3	NBS 4957 (1.8) [^{133}Ba]	AM RBZ (2 5) [Bi]	AM PMS (5) [^{208}Po int. std.]	AM RLZ (3.5) [^{228}Th int. std.]	AM TYS-1 (<5) [cf. ^{230}Th]	AM RLS (3.5) [cf. ^{230}Th]	AM TYS-1 (5) [cf. ^{226}Ra]
Lab 4	NBS 4957 (1.8) [^{133}Ba]	–	AM PMS (5) [^{208}Po int. std.]	– – – – – – – – – – – – – – – – thorium nitrate [^{228}Th int. std.]	[cf. ^{230}Th]	[cf. ^{230}Th]	'aged' ThO_2 [no sepn.]
Lab 5	NBS 4953-C (1.8) [^{133}Ba]	AM RBZ (2.3) [Bi]	AM RBZ (2.3) [^{208}Po]	– – – – – – – – – – – – – – – – – – AM TYS-1 (<5) [^{234}Th]			[cf. ^{226}Ra]
Lab 6	AM RAY-31 (5) [n.i.]	AM RBZ (1.9) [Bi]	AM PDZ (3.4) [^{208}Po]	USEPA 1838-3 (5.7) [^{234}Th]	–	–	–
Lab 7	NBS 4959 (1.3) [^{133}Ba]	AM RBZ (2.3) [n.i.]	–	CCRMP BL-5 (?) [n.i.]	–	–	–
Lab 8	AM RAY -31 (5) [^{133}Ba]	NEN NES-200F (5) – – – – – – – – – – – – – – – – [n.i.]	[n.i.]	NEN NES-316 (4.5) – [^{234}Th]			NEN NES-200F (5) [c.f. ^{226}Ra]

Abbreviations: AM, Amersham Corp.; IPL, Isotope Product Laboratories; NBS, National Bureau of Standards (U.S.); NEN, New England Nuclear Ltd.; USEPA, U.S. Environmental Protection Agency; n.i., not indicated.

Table B-15 – Results of analysis of variance, ^{238}U-series isotopes*

		^{230}Th	^{226}Ra	^{210}Pb	^{210}Po
UTS-1	Ratio	7.02	0.89	8.31 [0.8]	34.9
	$F_{.95}$	2.57	2.44	2.60 2.81	2.68
	f_b, f_w	6,21	7,23	6,20 5,17	5,21
UTS-2	Ratio	26.3	1.77	3.18 [0.8]	20.7
	$F_{.95}$	2.55	2.35	2.56	2.64
	f_b, f_w	6,22	7,29	5,28	5,23
UTS-3	Ratio	7.08	1.00	1.29	18.55
	$F_{.95}$	2.55	2.46	2.53	2.64
	f_b, f_w	6,22	7,22	6,23	5,23
UTS-4	Ratio	41.6 [5.58]	3.90	10.7	45.3
	$F_{.95}$	2.57 2.77	2.40	2.56	2.66
	f_b, f_w	6,21 5,18	7,25	6,21	5,22
UTS-5	Ratio	24.7	14.0	12.9	21.5
	$F_{.95}$	2.51	2.42	2.57	2.68
	f_b, f_w	6,24	7,24	6,21	5,21

**Ratio of 'between-set' to 'within-set' mean-square variance with degrees of freedom f_b and f_w, resp. (4).*
[], *value after removal of probable outlier set.*

Table B-16 – Results of analysis of variance, ^{232}Th-series isotopes*

		^{232}Th	^{228}Ra	^{228}Th
UTS-1	Ratio	2.92 [1.94]	7.32	2.34
	$F_{.95}$	2.61 3.18	3.71	3.05
	f_b, f_w	7,17 4,13	3,10	4,15
UTS-2	Ratio	3.14 [2.88]	33.9	17.8
	$F_{.95}$	2.58 3.11	3.59	3.01
	f_b, f_w	7,18 4,14	3,11	4,16
UTS-5	Ratio	6.12 [7.87]	13.52	16.4
	$F_{.95}$	2.61 3.18	3.06	3.05
	f_b, f_w	7,17 4,13	4,15	4,15

**Alpha spectrometry methodology only.*

Table B-17 – Results of Dixon r tests applied to laboratory mean results

Material	Isotope	Set	No. sets	Test ratio	Critical Value, r**	
					P = 10%	P = 5%
UTS-1	^{210}Pb	Lab-7	7	.63	.51	.57
UTS-1	^{228}Ra	Lab-5*	4	.90	.77	.82
UTS-2	^{210}Pb	Lab-7	7	.63	.51	.57
UTS-4	^{230}Th	Lab-3	7	.71	.51	.57

Test result overruled; set means of the other three sets were judged to be fortuitously close.
**Relevant probabilities are twice values for predesignated end of the set values (B2).*

Table B-18 – Consensus values and related statistical parameters for isotope activities in tailings reference materials

Isotope	Consensus value, [CL], Bq g^{-1}[a] (No. sets, No. values, RSD (%), $\overline{CV}$(%))			
	UTS-1	UTS-2	UTS-3	UTS-4
^{230}Th	3.6 [3.0 – 4.2] (7,28,21,10)	4.4 [3.3 – 5.5] (2,79,26,24)	11.3 [10.5 – 12.1] (7,29,9.5)	22.9[b] [20.3 – 25.5] (6,24,13.7)
^{226}Ra	3.67 [3.52 – 3.82] (8,31,9.7)	5.6 [6.2 – 6.0] (8,37,13,8)	13.3 [12.7 – 13.9] (8,30,11,6)	38.6 [36.2 – 40.9] (8,33,9,5)
^{210}Pb	3.25[b] [3.03 – 3.47] (6,23,9,7)	4.55[b] [4.36 – 4.75] (6,39,12,9)	12.6 [12.1 – 13.2] (7,30,9,7)	32.4 [29.6 – 35.3] (7,26,9,4)
^{210}Po	3.1 [2.7 – 3.5] (6,27,10,4)	4.4 [3.7 – 5.1] (6,29,14,6)	11.8 [10.8 – 12.9] (6,29,6,4)	30.8 [25.8 – 35.9] (6,28,14,4)
^{232}Th[c,d]	.68 [.59 – .77] (5,18,16,11)	.88 [.67 – 1.08] (5,19,24,17)	(.16 ± .04)	(.48 ± .06)
^{228}Ra	.68 [.47 – .89] (4,14,19,10)	1.0 [0.7 – 1.4] (4,15,17,6)	–	–
^{228}Th[d]	.71 [.58 – .84] (5,20,21,13)	.92 [.58 – 1.25] (5,21,29,11)	(.16 ± .04)	(.23 ± .04)
^{231}Pa[d]	(.21 ± .03)	(.37 ± .05)	(.70 ± 1.0)	(2.4 ± .3)

a. CL = statistical uncertainty range at 95% confidence level, RSD = relative standard deviation of individual results (%), $\overline{CV}$ = average within-laboratory rel. std. dev. (%).
b. Data are exclusive of an outlying set.
c. ^{232}Th results from alpha spectrometry only. ^{232}Th concs. by NAA are incorporated with chemical results.
d. Single laboratory results and uncertaintly estimates in brackets are for information only.

Table B-19 – Radioisotope consensus program performance on UTS-5 [DH-Ia/SiO_2, 49.4 ± .8% ore]

Isotope	Consensus, Bq g^{-1} Mean	95% CL		CI, ± %*	Bias Estimate %**	No. Sets	No. values	RSD %	$\overline{CV}$, %
^{230}Th	15.4	14.2	−16.7	8.5	−2.9	7	31	9.1	3.5
^{226}Ra	16.4	14.9	−17.8	8.8	+3.2	8	32	10.9	3.9
^{210}Pb	14.3	12.4	−16.1	13.0	−10.0	7	28	14	6.0
^{210}Po	13.6	11.4	−15.9	16.6	−14.2	6	27	15	7.0
^{232}Th(a)	1.94	1.72	−2.15	11.2	+6.7	8	25	14	6.4
^{232}Th(b)	1.96	1.59	−2.34	19.2	+7.8	5	18	16	7.9
^{228}Ra	1.80	1.14	−2.47	37.3	−1.1	5	20	27	15.3
^{228}Th	1.98	1.60	−2.37	19.5	+8.9	5	20	16	6.7
^{231}Pa	(.58)	(.55 – .61)		(5)	−22.	1	4	–	12.9

**one-sided 95% confidence interval, %.*

***$(x_c - x_p)/x_p$ where x_p and x_c are predicted and consensus values, resp. Uncertainties in the bias estimates due to concentration and half-life uncertainties of uranium and thorium resp. are approx. ±2.1 and ±5.3.*

(a) all data.

(b) α-spectrometry results only.